

## ENGINEERING CHANGE NOTICE

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## 633306

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# "FINAL REPORT FOR TANK 241-C-204, AUGER SAMPLES 95-AUG-022 AND 95-AUG-023

John M. Conner  
Westinghouse Hanford Company, Richland, WA 99352  
U.S. Department of Energy Contract DE-AC06-87RL10930


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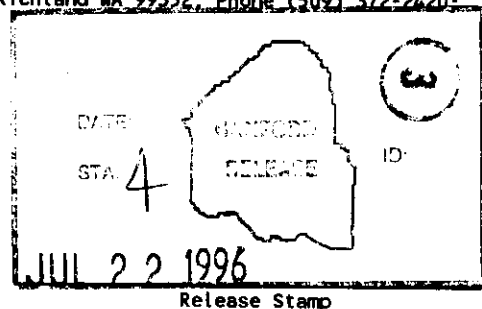
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1/22/90

90-Day Safety Screen Results for Tank 241-C-204, Auger Samples 95-AUG-022 and 95-AUG-023



**Westinghouse  
Hanford Company**

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P.O. Box 1970 Richland, WA 99352

**WHC-SD-WM-DP-115, REV. 1**

**ANALYTICAL SERVICES**

**FINAL REPORT FOR TANK 241-C-204,  
AUGER SAMPLES 95-AUG-022 AND 95-AUG-023**

**Project Coordinator: JOHN CONNER**

**Prepared for the U.S. Department of Energy  
Office of Environmental Restoration  
and Waste Management**

**by**

**Westinghouse Hanford Company  
Box 1970  
Richland, Washington**

**WHC-SD-WM-DP-115, REV. 1**

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Part I consists of pages 1 through 83 and pages ii, 2, 6, 10.1, 21, 25, 29, 35, 42, 46, and 70 were intentionally left blank.

Part II consists of pages 2-1 through 2-35 and pages 2-2, 2-5, 2-26, and 2-31 were intentionally left blank.

Part III consists of pages 3-1 through 3-51 and pages 3-2, 3-5, 3-13, and 3-17 were intentionally left blank.

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**NARRATIVE**

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**FINAL LABORATORY REPORTS FOR TANK 241-C-204,  
AUGER SAMPLES 95-AUG-022 AND 95-AUG-023**

**ANALYTICAL SUMMARY**

Two auger samples from tank 241-C-204 (C-204) were submitted to the 222-S Laboratories for safety screening analyses, consisting of differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and total alpha activity. Sampling and analysis requirements are presented in the Sampling and Analysis Plan (SAP) [1] (which was adapted with minimal changes from the original Tank Characterization Plan [2]. The Tank Characterization Plan was subsequently revised, and the sampling and analytical requirements were inadvertently deleted).

The DSC results for all samples from the tank exceeded the action limit of 481 J/g. Secondary analyses [total organic carbon (TOC) and adiabatic calorimetry], as negotiated with the safety program, were performed. The TOC results also exceeded the action limit of 3 weight percent. However, the moisture content of the samples is between 50 and 60%, and the results of adiabatic calorimetry testing indicated that the sample material would not support a propagating exothermic reaction. Primary and secondary analytical results were reported previously [3, 4] (these reports are also attached).

The 90-day report [4] indicated that additional heat capacity and DSC tests would be performed to further interpret the adiabatic calorimetry results. However, the laboratory has not been able to successfully perform heat capacity tests at this time. Therefore, this report is being issued without these results to provide a permanent record of the analytical work to date. Should the laboratory acquire the capability of performing these tests in the future, the safety program should consider whether further analysis of the remaining C-204 samples is worthwhile.

Organic speciation was also performed at the direction of the safety program. These analyses were performed by Battelle in the 329 Laboratory. These analyses were not performed to the controls in the SAP [1], but were performed to a Letter of Instruction (attached). The results indicate that the sample tested had an extremely high concentration of tributyl phosphate. Some dibutyl phosphate was also present. Other organics were present in only trace quantities.

**SCOPE**

This document contains all analytical results, raw data, photographs, and chain-of custody sheets for the tank C-204 auger samples (augers 95-AUG-022 and 95-AUG-023). Part I of this document consists of this abbreviated narrative describing the additional organic speciation work conducted by Battelle, photographs of the extruded augers, chain-of-custody

sheets, and all raw data not previously published (worklists for extrusion, sample preparation, TOC, and total alpha).

Part II consists of the 90-day report [4] in its entirety. The secondary safety screening results (TOC, adiabatic calorimetry, and additional DSC analyses) are presented and discussed in Part II. Part III consists of the 45-day report [3] in its entirety. The primary safety screening results (DSC, TGA, and total alpha) are described, and the raw data for the DSC and TGA analyses are provided.

## ORGANIC SPECIATION

At the request of the safety program, a sample from auger 95-AUG-023 was sent to Pacific Northwest National Laboratory (PNNL) for organic speciation. This work was funded and controlled outside the Sampling and Analysis Plan (see attached Letter of Instruction). The sample was analyzed for chelators, chelator fragments, low-molecular weight organic acids, and organically soluble carbon. Gas chromatography/mass spectrometry results indicate that the majority of the organic carbon was tributyl phosphate (TBP). Fully 33% of the sample was determined to be TBP. TIC/TOC and ion chromatography analyses (for water-soluble organics) were performed on the sample at PNNL as well. The results of these analyses were transmitted in a letter report (attached).

## REFERENCES

- [1] J. M. Conner, 1996, *Tank 241-C-204 Auger Sampling and Analysis Plan*, WHC-SD-WM-TSAP-089, Rev. 0.
- [2] R. D. Schreiber, 1995, *Tank 241-C-204 Tank Characterization Plan*, WHC-SD-WM-TP-307, Rev. 0.
- [3] J. M. Conner, 1995, *45-Day Safety Screen Results for Tank 241-C-204, Auger Samples 95-AUG-022 and 95-AUG-023*, WHC-SD-WM-DP-115, Rev. 0.
- [4] J. M. Conner, 1995, *90-Day Safety Screen Results for Tank 241-C-204, Auger Samples 95-AUG-022 and 95-AUG-023*, WHC-SD-WM-DP-115, Rev. 0A.

**WHC-SD-WM-DP-115, REV. 1**

**LETTER OF INSTRUCTION FOR ORGANIC SPECIATION**

WHC-SD-WM-DP-115, REV. 1

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P.O. Box 1970 Richland, WA 99352

July 31, 1995

9553938

Mr. J. A. Campbell  
Atomic and Molecular Chemistry  
Pacific Northwest Laboratory  
Post Office Box 999  
Richland, Washington 99352

Dear Mr. Campbell:

LETTER OF INSTRUCTION FOR TESTING OF SLUDGE SAMPLE FROM SINGLE-SHELL TANK  
241-C-204

This letter of instruction provides direction to Pacific Northwest Laboratory (PNL) for laboratory testing of a sludge sample from single-shell tank 241-C-204.

The intent of the testing by PNL is to determine the organic speciation of the sample. Testing should be focused on the major organic compounds typically found in Hanford waste streams (chelators, low molecular weight acids, normal paraffin hydrocarbons, tributyl phosphate). If these categories do not account for greater than 90% of the total organic carbon in the sample, then further direction from Westinghouse Hanford Company should be sought.

The sample will be shipped to Mr. Campbell of PNL. Funding for PNL for this project is provided by the Organic Safety Program under charge code N2E1D. Cost for the project is estimated at \$20 to 30K. For any funding questions, contact Mr. D. R. Johnson at 373-1747.

Detailed information regarding sample handling and analysis, and background information on tank 241-C-204 and the auger sampling event are attached. The signatures below signify acceptance of the terms listed here and in the attachments.

WHC-SD-WM-DP-116, REV. 1

CONDENSED STATEMENT OF WORK FOR TANK C-204 SAMPLE ORGANIC SPECIATION

Project contacts:	WHC - John M. Conner (373-2711) PNL - Jim A. Campbell (376-0899)
Shipping contacts:	WHC - Marty Martin PNL - Rick Steele
Cost Codes:	N2E1D
Number of samples:	1
Protocol to follow:	Pacific Northwest Laboratory (PNL) Level III
Deliverables:	Memo/letter report, including data and interpretation
Purpose of analysis:	Identify/quantify predominant organic compounds
Data to be validated?	No
Turnaround time	Four weeks (from receipt at PNL)
Analytical strategy	Focus on organics typically found in Hanford waste processing streams (chelates, low mol. wt. acids, normal paraffin hydrocarbons, tributyl phosphate. If these classes do not comprise > 90% of the total organic carbon of the sample, seek further direction from Westinghouse Hanford Company.



WHC-SD-WM-DP- 115, REV. 1

#### BACKGROUND INFORMATION ON THE 241-C-204 SAMPLE

Two auger samples were taken in May, 1995. Both augers were taken from riser 7 (only riser available). For both auger samples, a rag was caught in the auger. Visible rag material was segregated in the hot cell. One of the chemists reported seeing rag fibers in a sample from the tank. This suggests that the sample could be contaminated with rag material. However, by visual examination, the sample is largely tank waste solids (yellow and brown solids observed during extrusion).

Differential scanning calorimetry (DSC) results for samples from 241-C-204 exhibited exotherms well above the criterion of 481 J/g. Final values could not be obtained in many instances as the DSC scans did not return to baseline (exotherm still progressing at the limit of the test, 500 or 600 °C). An estimate of the average value is 1200 J/g. One sample will be submitted for adiabatic calorimetry to better characterize the exothermic nature of the sample.

Two samples were submitted for total organic carbon (TOC) analyses by hot persulfate oxidation (direct analysis). The average of the sample and duplicate results was 128,000 ug C/mL. This is almost 13 wt% TOC, uncorrected for moisture (moisture content was around 55% H<sub>2</sub>O, derived from thermogravimetric analysis results). Incidentally, the total inorganic carbon results averaged around 10,500 ug C/mL.

Some corroborating information from tanks 241-C-201 and 241-C-202 exists. Samples from these tanks also exhibited high exotherms (by DSC) and high TOC levels. As these tanks had very similar transfer histories, this supports the premise that the high exotherms and high TOC for 241-C-204 are partly due to organic material in the tank waste.

Dose rates for samples from tank 241-C-204 ranged from 125 to 350 mrad/hr (these samples would have been about 8 g, maximum. The sample being sent to Pacific Northwest Laboratory (PNL) is probably about 15 g). The dose for this sample will be listed on the chain of custody.

The sample which will be submitted to PNL for organic speciation is the remaining archive material from auger sample 95-AUG-023 from tank 241-C-204 (Westinghouse Hanford Company LABCORE sample number S95T000892, vial 7171).

**WHC-SD-WM-DP-115, REV. 1**

**LETTER REPORT: RESULTS OF ORGANIC SPECIATION**

WHC-SD-WM-DP-115, REV. 1

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WHC-SD-WM-DP-115, REV. 1

**Letter Report**  
**Tank 241-C-204 Analyses**

G.M. Mong  
J.A. Campbell

March 1996

Prepared for  
the U.S. Department of Energy  
under Contract DE-AC06-76RLO 1830

Pacific Northwest National Laboratory  
Richland, Washington 99352

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## Summary

This report summarizes the organic analyses of two auger samples taken from Tank 241-C-204 in May 1995. The major organic species present in the samples delivered to PNNL was tributyl phosphate (TBP), 0.18 g of carbon/g of wet weight. This corresponds to 0.33 g of TBP/g of wet weight sample as delivered to the hot cell. Relatively small amounts of dibutyl phosphate (DBP) were present in the water-soluble materials, representing about 2000  $\mu\text{g}$  of carbon/g of original tank sample. No monobutyl phosphate (MBP) was found to be present in the extracts presented for analysis. However, the assay for DBP and MBP may be incomplete; studies must be undertaken to determine the solubility limit of DBP and MBP in aqueous base to be certain that these materials are not left behind in the necessary ion-exchange step used to reduce the radioactivity of the final matrix material.

Acetate and formate were also present in the tank waste in very minor amounts (analysis by ion chromatography [IC]).

## Introduction

Two auger samples were taken from Tank 241-C-204 (Tank C-204) in May 1995. Both samples were taken from riser 7. Unfortunately, for both auger samples, a rag was caught in the auger. Visible rag material was segregated in the hot cell. One of the chemists reported seeing rag fibers in a sample from the tank which suggested that the sample might be contaminated with rag material. However, by visual examination, the sample was largely tank waste solids (yellow and brown solids observed during extrusion).

Westinghouse Hanford Company performed differential scanning calorimetry (DSC) for samples from Tank C-204. Results exhibited exotherms well above the criterion of 481 J/g. Final values could not be obtained in many instances as the DSC scans did not return to baseline. An estimate of the average value is 1200 J/g.

Two samples were also submitted for total organic carbon (TOC) analyses by hot persulfate oxidation (direct analysis). The average of the sample and duplicate results was 128,000  $\mu\text{g C/mL}$ . This is almost 13 wt% TOC, uncorrected for moisture (moisture content was approximately 55%  $\text{H}_2\text{O}$  derived from thermogravimetric analysis results). The total inorganic carbon results averaged 10,500  $\mu\text{g C/mL}$ .

Samples were submitted to the Advanced Organic Analytical Methods Group at PNNL for organic analysis. The samples from Tank 204-C were analyzed in the 329 laboratory for chelators, chelator fragments, low-molecular weight organic acids, and organically soluble carbon. The results indicated the majority of the organic carbon consisted of tributyl phosphate (TBP).

## Experimental

Approximately 15 g of total tank sample were taken in the 325 West hot cell. Two duplicate samples, approximately 1.8-g samples each, were made basic with 6N NaOH and eluted through ion exchange beds (Dowex 50-X8, Na<sup>+</sup> form) to reduce radioactivity (Campbell et al., 1994). The samples were then surveyed for dose rate and released from the hot-cell facility.

Two 1-g samples of the original tank sample were stirred with water to dissolve salts and then extracted with measured volumes of methylene chloride. These samples were released from the hot-cell facility after survey for dose rate. The bench sheets from the hot-cell technician reveal that a "thin floating layer" was observed in at least one sample after dissolution with water.

Aliquots of the aqueous extracts were taken to dryness (2 mL of the total aqueous extract; representing approximately 100 mg of the original tank matrix) for analysis of organic acids. These samples were treated with BF<sub>3</sub>/methanol complex (2 mL) for 1 h at 100° C to derivatize potential chelator components such as ethylenediaminetetraacetic acid (EDTA), nitrilotriacetic acid (NTA), and N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA).

Samples of the aqueous extract were deemed low enough in radioactivity to allow benchtop ion chromatographic separation for the analysis of formate, acetate, and oxalate components. Samples (20 µL) were introduced into the ion chromatography (IC) system and eluted from an AS-11 (Dionex) column using a ramped sodium hydroxide eluent (0.5 mM to 38 mM NaOH). An additional separation using 25 mM of NaOH and an AS-4A (Dionex) system was employed for the quantification of dibutyl phosphate (DBP).

The methylene chloride extracts were dried with a bed of sodium sulfate to remove residual aqueous radioactive components, taken to a minimum volume (300 µL) using a gentle stream of dry nitrogen, and reconstituted to a known volume (5 mL) with methylene chloride. Dilutions of this known volume were used to analyze for extractable organic components (e.g., TBP).

For quantitation results, TBP 99+% (Aldrich Chemical Co; lot # 06828CX) and butyl phosphate, tech. (Pfaltz and Bauer; lot # 040927) were used. The TBP was assumed to be adequate for a comparison standard, based on analytical results from the manufacturer. Butyl phosphate, tech. grade, has been found to be an essentially 50:50 mix of monobutyl phosphate (MBP) and DBP based upon its reaction with diazomethane and subsequent gas chromatography analysis. Additionally, we have examined butyl phosphate by IC to ascertain the approximate



distribution of butyl phosphates in the liquid. Since these materials are in dynamic equilibrium, any quantitation for these two components should be considered to be approximate.

Weights of materials were determined in the laboratory using a calibrated Mettler AC 100 balance (sensitivity = 0.1 mg). Dilution volumes were done using Eppendorf 1 mL and 0.1 mL adjustable pipettes that were previously calibrated using the weighed volumes of water delivered from the pipettes. Class A volumetrics were used for bulk dilutions.

Mass spectrometric data were obtained on low resolution gas chromatography/mass spectrometry (GC/MS) systems (Hewlett-Packard 5888 and 5885) that were tuned with PFTBA before use. Standards of TBP were run concurrently with the samples to determine relative response. Duplicate samples were run with each analysis. Additional GC/MS data were obtained using a HP 5970 MSD system, tuned to PFTBA before use. Quantitative data were not obtained from the HP 5970 instrument.

Aqueous extract samples were treated with an unquantified excess of ethereal diazomethane (formed from base treatment of N-methyl-N-nitrosourea) for estimation of DBP by GC/MS analysis.

Ion chromatographic responses were obtained using a Dionex conductivity detector system and acetic acid-glacial (Mallinckrodt, lot 8817) and formic acid 95-97% (Aldrich, Lot # 04324EV) as primary standards.

The TOC/total inorganic carbon (TIC) analyses performed in the 325 laboratory were checked versus glucose (Kodak lot #B1F) and calcium carbonate (lot #N261) as check standards. The TOC/TIC measurements were done in duplicate and with spiked check samples.

## Results and Discussion

The aqueous extracts obtained from Tank C-204 did not reveal any quantifiable amount of the typical chelator components associated with tank wastes (EDTA, NTA, HEDTA, or citric acid). The IC analysis revealed small (less than 100 µg/g sample) amounts of acetate, formate, and oxalate to be present in the matrix material. Trace amounts of butyric acid, toluene, and benzoic acid were confirmed to be present in the matrix from analysis of the BF<sub>3</sub>/methanol complex derivatized samples. None of these were quantified at this time.

Gas chromatographic/mass spectrometric (GC/MS) analysis of the methylene chloride extracts revealed TBP to be the major carbon species present. The value for TBP obtained from GC/MS analysis of the methylene chloride extracts averages 0.18 g of carbon/g of wet weight sample (at time of sampling in the hot cell). This corresponds to 0.33 g of TBP/g of sample as delivered to the hot cell. This unexpected result was verified by an additional duplicate GC/MS analysis. The observation of a visible floating layer in the hot-cell workup is supported by the high value obtained for TBP. For very concentrated samples of the TBP extracts obtained from Tank C-204, trace amounts of normal paraffin hydrocarbons were also observed in the GC/MS analysis. These were present in such minor amounts (compared to TBP) that a quantification was not attempted.

Ion chromatographic analysis of the aqueous extracts of Tank C-204 revealed the presence of DBP. Quantification was done using a 25-mM NaOH eluent and an AS-4A (Dionex) column. The DBP concentration was found to be approximately 2500 µg of carbon/g of sample by this method. Two samples of the aqueous matrix were acidified with hydrochloric acid, taken to dryness and treated with an ethereal solution of diazomethane (above) to allow GC quantification of dibutyl phosphate as dibutylmethyl phosphate. The result obtained from this analysis gave a value of DBP of 2000 µg of carbon/g of sample as received. It is interesting to note that in neither the IC data nor the diazomethane derivatization/GC analyses was MBP observed. The IC data suggest that the level of inorganic phosphate is in similar concentration to that observed for DBP; the intermediate degradation product (monobutyl phosphate) being absent.

The TOC analysis of the waste matrix by hot persulfate oxidation revealed about 60,000 µg oxidizable carbon /g of sample to be present in the material (preliminary result). This is in line with the observations of D. Baldwin (1994) related to other TOC data obtained with TBP. Baldwin interprets his results to indicate that the hot persulfate oxidation does not give complete or quantitative results with TBP. The value obtained for TOC after ion exchange was about 500 µg

C/g of sample, indicating that this value is also representative of incomplete oxidation of dibutyl phosphate.

## Conclusion

The major organic species present in the samples of Tank C-204 matrix delivered to PNNL is TBP. Fully 0.18 g of carbon/g of wet weight sample is accounted for as TBP. This corresponds to 0.33 g of TBP/g of wet weight sample as delivered to the hot cell. Relatively small amounts of DBP appear in the water soluble materials, representing about 2000  $\mu\text{g}$  of carbon/g of original tank sample. Quite surprisingly, no MBP was found to be present in the extracts presented for analysis. The potential for incomplete assay of DBP and MBP in the aqueous phase exists; studies must be undertaken to determine the solubility limit of DBP and MBP in aqueous base to be certain that these materials are not left behind in the necessary ion-exchange step used to reduce the radioactivity of the final matrix material.

Acetate and formate appear to be present in the tank waste in very minor amounts (analysis by IC).

## References

- Campbell, J. A., R. M. Bean, G. M. Mong, S. A. Clauss, R. B. Lucke, B. D. Lerner, K. A. Grant, R. Steele, V. Hoopes, and J. Rau. 1994. *Flammable Gas Safety Program. Analytical Methods Development: FY 1993 Progress Report*. PNL-9062, Pacific Northwest Laboratory, Richland, Washington.
- Baldwin, D.L., R.W. Stromatt, and W.I. Winters. 1994. In: *Spectrum '94, Nuclear and Hazardous Waste Management International Topical Meeting*, Atlanta, Ga, June 1994, PNL-SA-23718, Pacific Northwest National Laboratory, Richland, Wa.

**WHC-SD-WM-DP-115, REV. 1**

**SAMPLE DATA SUMMARY**

**WHC-SD-WM-DP-115, REV. 1**

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Analytical Summary Table - Final Report  
C-204

CORE NUMBER: n/a  
SEGMENT #: 95-AUG-022

SEGMENT PORTION: U Upper Half of Segment

Sample#	R A#	Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
S95T000878		DSC Exotherm using Mettler	Joules/g	103.7	n/a	>1.96e+02	>542.0	n/a	n/a	n/a	n/a	n/a
S95T000878		DSC Exotherm Dry Calculated	Joules/g Dry	n/a	n/a	>4.46e+02	>1234.0	n/a	n/a	n/a	1.00e-04	n/a
S95T000878		% Water by TGA using Mettler	%	99.29	n/a	58.32	50.44	54.38	14.5	n/a	n/a	n/a
S95T000879	F	Alpha of Digested Solid	uCi/g	90.54	<2.81e-03	6.43e-03	1.45e-02	1.05e-02	77.1	61.90	7.00e-03	73.8

L Lower Half of Segment: L Lower Half of Segment

Sample#	R A#	Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
S95T000881		DSC Exotherm using Mettler	Joules/g	103.7	n/a	>2.87e+02	>33.4	n/a	n/a	n/a	n/a	n/a
S95T000881		DSC Exotherm Dry Calculated	Joules/g Dry	n/a	n/a	>6.47e+02	>76.1	n/a	n/a	n/a	1.00e-04	n/a
S95T000881		% Water by TGA using Mettler	%	99.29	n/a	55.02	56.39	55.70	2.46	n/a	n/a	n/a
S95T000882	F	Alpha of Digested Solid	uCi/g	90.54	<2.81e-03	2.34e-02	1.21e-02	1.78e-02	63.7	n/a	7.00e-03	33.3
S95T000961		TIC by Acid/Coulometry	ug/g	90.00	2.600	9.36e+03	8.13e+03	8.74e+03	14.1	77.30	5.000	n/a
S95T000961		TOC by Persulfate/Coulometry	ug/g	92.67	35.70	1.48e+05	1.30e+05	1.39e+05	12.9	119.0	80.00	n/a

22

WHC-SD-WM-DP-115, REV. 1

Analytical Summary Table - Final Report  
C-204

CORE NUMBER: n/a  
SEGMENT #: 95-AUG-023

SEGMENT PORTION: Facie

Sample#	R	A#	Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
S95T000966			DSC Exotherm using Mettler	Joules/g	102.8	n/a	0.00e+00	0.00e+00	0.00e+00	n/a	n/a	n/a	n/a
S95T000966			DSC Exotherm Dry Calculated	Joules/g Dry	n/a	n/a	0.00e+00	0.00e+00	0.00e+00	n/a	n/a	n/a	n/a

W Whole Segment: W Whole Segment

Sample#	R	A#	Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
S95T000890			DSC Exotherm using Mettler	Joules/g	107.2	n/a	4.00e+02	279.6	339.8	35.4	n/a	n/a	n/a
S95T000890			DSC Exotherm Dry Calculated	Joules/g Dry	n/a	n/a	9.52e+02	665.7	808.9	35.4	n/a	1.00e-04	n/a
S95T000890			% Water by TGA using Mettler	%	99.76	n/a	59.92	56.08	58.00	6.62	n/a	n/a	n/a
S95T000891	F		Alpha of Digested Solid	uCi/g	105.7	1.40e-02	5.11e-02	5.19e-02	5.15e-02	1.55	n/a	7.00e-03	17.6
S95T000963			TIC by Acid/Coulometry	ug/g	90.00	2.600	1.38e+04	1.07e+04	1.22e+04	25.3	n/a	5.000	n/a
S95T000963			TOC by Persulfate/Coulometry	ug/g	92.67	35.70	9.18e+04	1.44e+05	1.18e+05	44.3	n/a	80.00	n/a

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WHC-SD-WM-DP-115, REV. 1



**WHC-SD-WM-DP-115, REV. 1**

**CHAIN OF CUSTODY FORMS**

WHC-SD-WM-DP-115, REV. 1

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COPY

## CHAIN-OF-CUSTODY RECORD FOR AUGER SAMPLING

COPY

(1) Shipment Number 200 W-08-TF (2) Sample Number 95-AUG-023 (3) Supervisor JAMES SICKELS  
 (4) Tank C-204 (5) Riser 7 WEST (6) Cask Serial Number \* C 1053

Radiation Survey Data:		(7) FIELD	(31) LABORATORY	(8) Shipment Description	
Over Top Dose Rate	<u>&lt;5 mR/hr</u>		<u>20.5 mR/hr</u>	A. Work Package Number	<u>ES-95-00012/0</u>
Side Dose Rate	<u>&lt;5 mR/hr</u>		<u>20.5 mR/hr</u>	B. Cask Seal Number	<u>1039</u>
Bottom Dose Rate	<u>&lt;5 mR/hr</u>		<u>20.5 mR/hr</u>	C. Date and Time Sample	<u>5-2-95 11:55 AM</u>
Smearable Contamination	<u>&lt;20 dpm/100cm<sup>2</sup></u> (Alpha)		<u>&lt;20 dpm/100cm<sup>2</sup></u> (Alpha)	Removed from Tank	
	<u>&lt;1K dpm/100cm<sup>2</sup></u> (Beta-Gamma)		<u>&lt;1K dpm/100cm<sup>2</sup></u> (Beta-Gamma)	D. Expected Liquid Content	<u>10%</u>
RCT <u>[Signature]</u>	(Signature)	RCT <u>[Signature]</u>	(Signature)	E. Expected Solid Content	<u>90%</u>
				F. Dose Rate Through Drill String	<u>6 mR/hr</u>
				G. Expected Sample Length	<u>15"</u>

(9) INFORMATION (Include statement of laboratory tests to be performed.)

(10) Field Comments

(32) Laboratory Comments

(11) Point of Origin <u>C-204 R<sup>#</sup>7</u>	(12) Destination <u>2025 LAB</u>	(13) Sender Name (Sign and PRINT) <u>JAMES SICKELS James Sickels</u>	(14) Date/Time <u>5-3-95/1012</u>	(15) Sender Comments
(17) Relinquished By (Sign and PRINT) <u>James Sickels James Sickels</u>	(18) Received By (Sign and PRINT) <u>Charles E. Byrd C.E. Byrd</u>	(19) Date/Time <u>5-3-95 1342</u>	(20) Receiver Comments	
(21) Relinquished By (Sign and PRINT) <u>James Sickels</u>	(22) Received By (Sign and PRINT) <u>C.E. Byrd</u>	(23) Date/Time <u>5-3-95/1430</u>	(24) Receiver Comments	
(25) Relinquished By (Sign and PRINT)	(26) Received By (Sign and PRINT)	(27) Date/Time	(28) Receiver Comments	

(10) Seal Intact Upon Release?

☒ Yes ☐ No

(29) Seal Intact Upon Receipt?

☒ Yes ☐ No

(30) Seal Data Consistent with this Record?

Shipment No.

☒ Yes ☐ No

Cask Seal No.

☒ Yes ☐ No

Sample No.

☒ Yes ☐ No

WHC-SD-WM-OP-115 REV. 1

COPY

2

## CHAIN-OF-CUSTODY RECORD FOR AUGER SAMPLING

COPY

(1) Shipment Number 200W-08-T.F. (2) Sample Number 95-AUG-022 (3) Supervisor James Sickels  
 (4) Tank C-204 (5) Floor # 7 ENT (6) Cask Serial Number # 10148 C

## Radiation Survey Data:

(7) FIELD

(31) LABORATORY

(8) Shipment Description

Over Top Dose Rate

&lt; 5 mR/hr

20.5 mR/hr

A. Work Package Number

ES-95-00012/0

Side Dose Rate

&lt; 5 mR/hr

20.5 mR/hr

B. Cask Seal Number

1040

Bottom Dose Rate

&lt; 5 mR/hr

20.5 mR/hr

C. Date and Time Sample

5-2-95 10:30 AM

Smearable Contamination

< 20 dpm/100cm<sup>2</sup>< 20 dpm/100cm<sup>2</sup>

Removed from Tank

(Alpha)

(Alpha)

D. Expected Liquid Content

10%

< 1K dpm/100cm<sup>2</sup>< 1K dpm/100cm<sup>2</sup>

E. Expected Solid Content

90%

(Beta-Gamma)

(Beta-Gamma)

F. Dose Rate Through Drill String

3.5 mR/hr

RCT\*

RCT\*

(Signature)

(Signature)

G. Expected Sample Length

15"

(9) INFORMATION (Include statement of laboratory tests to be performed.)

(10) Field Comments

(32) Laboratory Comments

WHG-SD-WM-OP. 1/2 REV.

(11) Point of Origin

(12) Destination

(13) Sender Name (Sign and PRINT)

(14) Date/Time

(15) Sender Comments

C-204 R<sup>#</sup>7

2225 LAB

JAMES SICKELS

5-3-95/1312

(17) Relinquished By (Sign and PRINT)

(18) Received By (Sign and PRINT)

(19) Date/Time

(20) Receiver Comments

James Sickels

Charles E. Dugas

5-3-95/1312

(21) Relinquished By (Sign and PRINT)

(22) Received By (Sign and PRINT)

(23) Date/Time

(24) Receiver Comments

James R. Dugas

E. E. Dugas

5-3-95/1422

(25) Relinquished By (Sign and PRINT)

(26) Received By (Sign and PRINT)

(27) Date/Time

(28) Receiver Comments

(16) Seal Intact Upon Release?

(29) Seal Intact Upon Receipt?

(30) Seal Data Consistent with this Record?

☒ Yes ☐ No☒ Yes ☐ No

Shipment No.

☒ Yes ☐ No

Cask Seal No.

☒ Yes ☐ No

Sample No.

☒ Yes ☐ No

**WHC-SD-WM-DP-115, REV. 1**

**PHOTOGRAPHS**

WHC-SD-WM-DP-115, REV. 1

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5/4/95

95-AUG-022

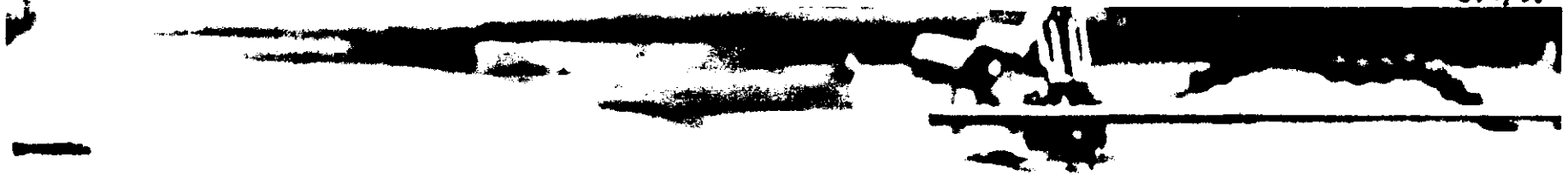
C-204

WHC-SD-WM-DP-115

C-204

95-AUG-022

5/4/95



WHC-SD-WM-DP-115, REV. 1

31 BEST AVAILABLE COPY



5/5/95

95-AUG-023

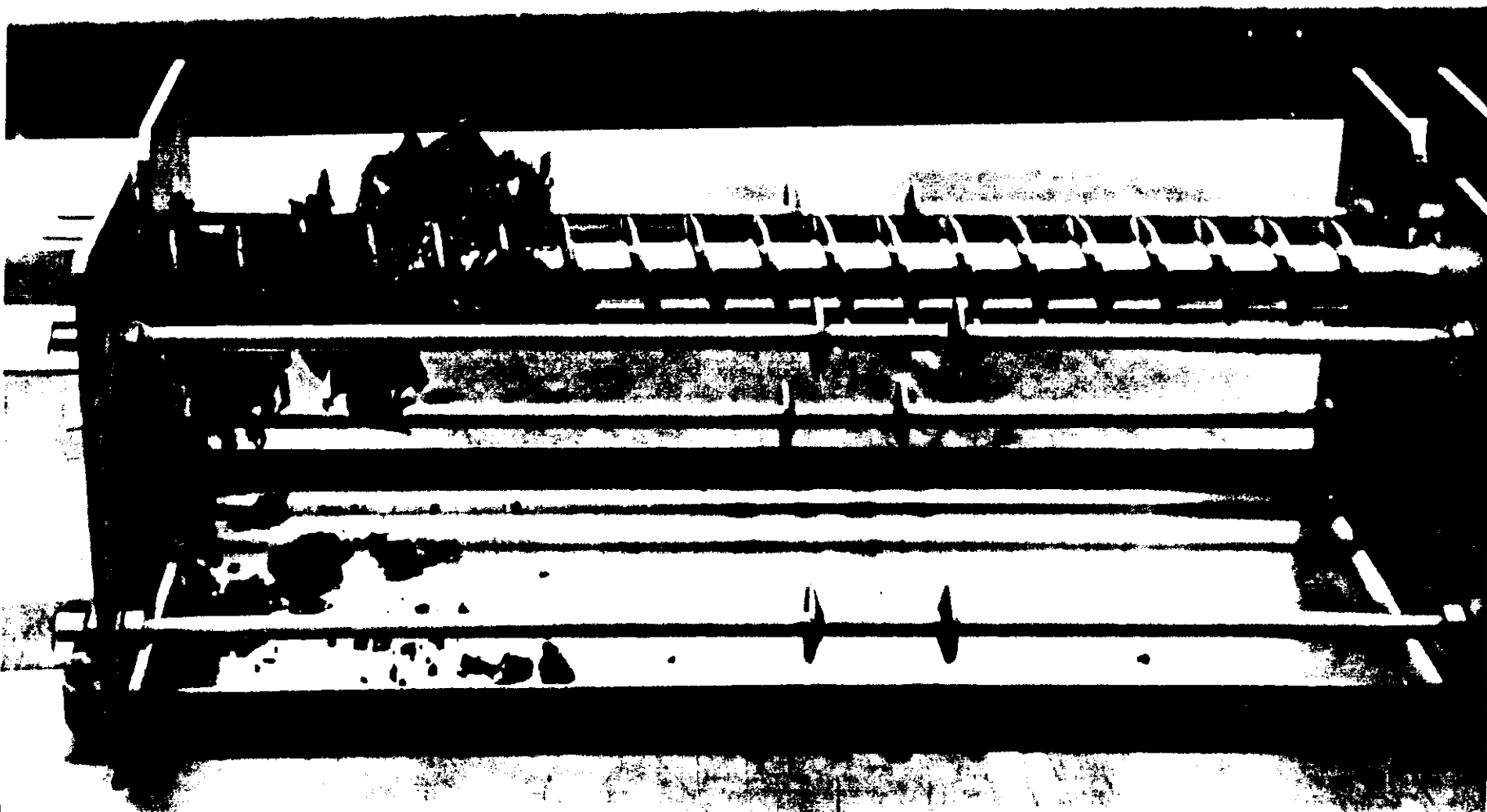
C-204



C-204

95-AUG-023

5/5/95



WHC:SD-WM-DP-115, REV. 1

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WHC-SD-WM-DP-115, REV. 1

## SAMPLE HANDLING

**WHC-SD-WM-DP-115, REV. 1**

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WHC-SD-WM-DP-115, REV. 1  
**LABCORE Data Entry Template for Worklist# 1335**

Analyst: EC Instrument: BA000 Book # NA

Method: LO-160-103 Rev/Mod A-7

Worklist Comment: C-204 95-AUG-022 Riser 7 East Extrusion

GROUP	PROJECT	S	TYPE	SAMPLE#	R	A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1	INSTCHK01				EXTRUD01	SOLID	<u>20</u>	<u>19.99</u>	<u>N/A</u>	
		2	INSTCHK02				EXTRUD01	SOLID	<u>500</u>	<u>500.00</u>	<u>N/A</u>	
95000069		3	SAMPLE	S95T000876	0		DLIQVOL1	SOLID	<u>N/A</u>	<u>0</u>		mL
95000069		4	SAMPLE	S95T000876	0		DLIQWT01	SOLID	<u>N/A</u>	<u>0</u>		g
95000069		5	SAMPLE	S95T000876	0		EST.G/ML	SOLID	<u>N/A</u>	<u>0</u>		g/mL
95000069		6	SAMPLE	S95T000876	0		EXTRUD01	SOLID	<u>N/A</u>	<u>complete</u>		
95000069		7	SAMPLE	S95T000876	0		LLIQWT01	SOLID	<u>N/A</u>	<u>0</u>		g
95000069		8	SAMPLE	S95T000876	0		NOTEBOOK	SOLID	<u>N/A</u>	<u>WHC-10-114.2</u>		
95000069		9	SAMPLE	S95T000876	0		SLDVOL01	SOLID	<u>N/A</u>	<u>158</u>		mL
95000069		10	SAMPLE	S95T000876	0		SLDWT-01	SOLID	<u>N/A</u>	<u>158</u>		g
95000069		11	SAMPLE	S95T000876	0		ORGVOL01	SOLID	<u>N/A</u>	<u>0</u>		mL

**Final page for worklist # 1335**

EC 5-5-95  
Analyst Signature Date

EC 5-5-95  
Analyst Signature Date

Data Entry Comments:

Reviewed by RK Fisher  
5/5/95

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number,  
R = Replicate Number, A = Aliquot Code.

# LABCORE Data Entry Template for Worklist# 1336

Analyst: EC Instrument: BA000 Book # NA

Method: LO-160-103 Rev/Mod A-7

Worklist Comment: C-204 95-AUG-023 Riser 7 West Extrusion

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 INSTCHK01			EXTRUD01	SOLID	<u>20g</u>	<u>19.99</u>	<u>N/A</u>	
		2 INSTCHK02			EXTRUD01	SOLID	<u>500g</u>	<u>499.99</u>	<u>N/A</u>	
95000069		3 SAMPLE	S95T000877	0	DLIQVOL1	SOLID	<u>N/A</u>	<u>0</u>		mL
95000069		4 SAMPLE	S95T000877	0	DLIQWT01	SOLID	<u>N/A</u>	<u>0</u>		g
95000069		5 SAMPLE	S95T000877	0	EST.G/ML	SOLID	<u>N/A</u>	<u>0</u>		g/mL
95000069		6 SAMPLE	S95T000877	0	EXTRUD01	SOLID	<u>N/A</u>	<u>complete</u>		
95000069		7 SAMPLE	S95T000877	0	LLIQWT01	SOLID	<u>N/A</u>	<u>0</u>		g
95000069		8 SAMPLE	S95T000877	0	NOTEBOOK	SOLID	<u>N/A</u>	<u>WIK-N-1142</u>		
95000069		9 SAMPLE	S95T000877	0	SLDVOL01	SOLID	<u>N/A</u>	<u>134</u>		mL
95000069		10 SAMPLE	S95T000877	0	SLDWT-01	SOLID	<u>N/A</u>	<u>134</u>		g
95000069		11 SAMPLE	S95T000877	0	ORGVOL01	SOLID	<u>N/A</u>	<u>0</u>		mL

Final page for worklist # 1336

EC 5-5-95  
Analyst Signature Date

EC 5-5-95  
Analyst Signature Date

Data Entry Comments:

Reviewed by RK Fuller  
8/8/95

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

# LABCORE Data Entry Template for Worklist# 1346

Analyst: dy Instrument: BA000 Book # \_\_\_\_\_

Method: LO-160-103 Rev/Mod A7

Worklist Comment: C-204 95 AUG-022 ARCHIVE

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1	INSTCHK01		EXTRUD01	SOLID	20	19.986	N/A	
		2	INSTCHK02		EXTRUD01	SOLID	500	477.8	N/A	
95000069	C-204	3	SAMPLE	S95T000885	0 X	ARCHIV01	SOLID	N/A	36.43	g

## Final page for worklist # 1346

A. Mag 5-5-95  
Analyst Signature Date

A. Mag 5-5-95  
Analyst Signature Date

Archive sample is vial # 7168 (40ml / 20ml)  
dy 5-5-95  
Parent jar #6789 was used in process.

Data Entry Comments: Reviewed by RK Miller 5/8/95

# LABCORE Data Entry Template for Worklist# 1362

Analyst: ABB Instrument: BA000 Book # NA

Method: LO-160-103 Rev/Mod A-7

Worklist Comment: C-204 95-AUG-023 RISER # 7 ARCHIVE

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 INSTCHK01			EXTRUD01	SOLID	<u>26</u>	<u>19.99</u>	<u>N/A</u>	
		2 INSTCHK02			EXTRUD01	SOLID	<u>500</u>	<u>444.74</u>	<u>N/A</u>	
95000069	C-204	3 SAMPLE	S95T000892	0 X 6750	ARCHIV01 7/71	SOLID	<u>N/A</u>	<u>24.49</u>		g

Final page for worklist # 1362

Allen B. Bandy 5-9-95  
Analyst Signature Date

\_\_\_\_\_  
Analyst Signature Date

Data Entry Comments:

reviewed by  
Allen B. Bandy  
5-9-95

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.



LABCORE Data Entry Template for Worklist#

1868

Analyst: ABC Instrument: BA000 Book # \_\_\_\_\_

Method: LO-160-103 Rev/Mod A-7

Worklist Comment: C-204 95-aug-023 Shipment to PNL

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 INSTCHK01			EXTRUD01	SOLID	<u>20</u>	<u>19.99</u>	<u>N/A</u>	
		2 INSTCHK02			EXTRUD01	SOLID	<u>500</u>	<u>499.74</u>	<u>N/A</u>	
95000069	C-204	3 SAMPLE	S95T000892	O P 7/7/	SHIP-PNL	SOLID	<u>N/A</u>	<u>20.7</u> <u>24.4</u>		

RK7u  
7/20/95

Final page for worklist #

1868

ABCampbell 7/20/95  
Analyst Signature Date

Alan B Campbell 7/20/95  
Analyst Signature Date

Reviewed by JAR B. Bay 7-20-95

Data Entry Comments:

JAR # 7171 contains C-204 95-AUG-023 lower half RK7 7/20/95  
solids/rag material.

WHC-SD-WM-DP-115, REV. 1

## SAMPLE PREPARATIONS

WHC-SD-WM-DP-115, REV. 1

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# LABCORE Data Entry Template for Worklist# 1359

Analyst:

*[Signature]*

Instrument:

FUS01 AL11006

Book #

N/A

Method: LA-549-141 Rev/Mod

C-3

Worklist Comment: C-204 FUSION - 878->879, 881->882

GROUP	PROJECT	S	TYPE	SAMPLE#	R	A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1	BLNK-PREP				FUSION01	SOLID	250ml		N/A	g/L
95000069	C-204	2	SAMPLE	S95T000879	0	F	FUSION01	SOLID	N/A	2.0536		g/L
				.5134 → 250ml								
95000069	C-204	3	DUP	S95T000879	0	F	FUSION01	SOLID	2.0536	2.1156	N/A	g/L
				.5289 → 250ml								
95000069	C-204	4	SAMPLE	S95T000882	0	F	FUSION01	SOLID	N/A	2.0304		g/L
				.5076 → 250ml								
95000069	C-204	5	DUP	S95T000882	0	F	FUSION01	SOLID	2.0304	2.0676	N/A	g/L
				.5169 → 250ml								

Final page for worklist # 1359

Analyst Signature

Date

Analyst Signature

Date

Data Entry Comments:

Scale calibrated 200/100/100. < 1% solids.

20ml HCl, 3m Rad/hr by Jim Evans

Bernard Giffin II

5-11-95

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

# LABCORE Data Entry Template for Worklist# 1360

Analyst: QAM Instrument: FUS01 AL 11066 Book # N/A

Method: LA-549-141 Rev/Mod C-23

Worklist Comment: C-204 FUSION - 890->891

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 BLNK-PREP			FUSION01	SOLID	<u>250ml</u>		N/A	g/L
95000069	C-204	2 SAMPLE	S95T000891	0 F	FUSION01	SOLID	N/A	<u>2.2284</u>		g/L
			<u>.5571 → 250ml</u>							
95000069	C-204	3 DUP	S95T000891	0 F	FUSION01	SOLID	<u>2.2284</u>	<u>2.1852</u>	N/A	g/L
			<u>.5463 → 250ml</u>							

Final page for worklist # 1360

Yeresa Murphy 5-17-95  
Analyst Signature Date

Bertrand Giffon II 5-22-95  
Analyst Signature Date

Data Entry Comments:

30% acids  
Scale calibrated 200/100/100. Sample & dup are unclear,  
dark & murky. Was asked to submit anyway. 20 ml  
HCl. Data rate was 50mad/m. NPT was Scott Ganderback.

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number,  
R = Replicate Number, A = Aliquot Code.

BBI 5-22-95  
44

**WHC-SD-WM-DP-115, REV. 1**

**INORGANIC ANALYSES**

**WHC-SD-WM-DP-115, REV. 1**

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# LABCORE Data Entry Template for Worklist# 1570

Analyst: BDV Instrument: DSC0 1 Book # —

Method: LA-514-113 Rev/Mod —

Worklist Comment: Calculated dry DSC for C-204. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
95000069	C-204	1 SAMPLE	S95T000966	0	DSC-02	SOLID	N/A	Ø		Joules/g Dry
95000069	C-204	2 DUP	S95T000966	0	DSC-02	SOLID	Ø	Ø	N/A	Joules/g Dry

**Final page for worklist # 1570**

Data entered + verified by  
Blandina Valenzuela 6/13/95  
Analyst Signature Date

Analyst Signature Date

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.



# LABCORE Data Entry Template for Worklist# 1489

Analyst: KRM Instrument: TOC01 Book # 15N12F TIC  
16N12 TOC  
Method: LA-342-100 Rev/Mod A-D <sup>AD</sup> 6-8-95  
Worklist Comment: @TICTOC1 C-204 KRM

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 BLNK			@TICTOC1 TIC-02	SOLID	<u>1</u>	<u>2.6</u>	<u>N/A</u>	ug/g
		1 BLNK			@TICTOC1 TOC-02	SOLID	<u>1</u>	<u>35.7</u>	<u>N/A</u>	ug/g
		2 STD			@TICTOC1 TIC-02	SOLID	<u>6.00E<sup>2</sup></u>	<u>5.40E<sup>2</sup></u>	<u>N/A</u>	ug/g
		2 STD			@TICTOC1 TOC-02	SOLID	<u>3.00E<sup>3</sup></u>	<u>2.78E<sup>3</sup></u>	<u>N/A</u>	ug/g
95000069	C-204	3 SAMPLE	S95T000961	0	@TICTOC1 TIC-02	SOLID	<u>N/A</u>	<u>9.36E<sup>3</sup></u>	<u>5.0</u>	ug/g
95000069	C-204	3 SAMPLE	S95T000961	0	@TICTOC1 TOC-02	SOLID	<u>N/A</u>	<u>1.48E<sup>5</sup></u>	<u>80.0</u>	ug/g
95000069	C-204	4 DUP	S95T000961	0	@TICTOC1 TIC-02	SOLID	<u>9.36E<sup>3</sup></u>	<u>8.13E<sup>3</sup></u>	<u>N/A</u>	ug/g
95000069	C-204	4 DUP	S95T000961	0	@TICTOC1 TOC-02	SOLID	<u>1.48E<sup>5</sup></u>	<u>1.30E<sup>5</sup></u>	<u>N/A</u>	ug/g
95000069	C-204	5 SPK	S95T000961	0	@TICTOC1 TIC-02	SOLID	<u>100</u>	<u>77.3</u>	<u>N/A</u>	ug/g
95000069	C-204	5 SPK	S95T000961	0	@TICTOC1 TOC-02	SOLID	<u>100</u>	<u>119.0</u>	<u>N/A</u>	ug/g
95000069	C-204	6 SAMPLE	S95T000963	0	@TICTOC1 TIC-02	SOLID	<u>N/A</u>	<u>1.38E<sup>4</sup></u>	<u>5.0</u>	ug/g
95000069	C-204	6 SAMPLE	S95T000963	0	@TICTOC1 TOC-02	SOLID	<u>N/A</u>	<u>9.18E<sup>4</sup></u>	<u>80.0</u>	ug/g
95000069	C-204	7 DUP	S95T000963	0	@TICTOC1 TIC-02	SOLID	<u>1.38E<sup>4</sup></u>	<u>1.07E<sup>4</sup></u>	<u>N/A</u>	ug/g
95000069	C-204	7 DUP	S95T000963	0	@TICTOC1 TOC-02	SOLID	<u>9.18E<sup>4</sup></u>	<u>1.44E<sup>5</sup></u>	<u>N/A</u>	ug/g

Final page for worklist # 1489

[Signature] 6-4-95  
Analyst Signature Date

[Signature] 6-6-95  
Analyst Signature Date

Reviewed RW Schaefer 6-6-95

Data Entry Comments:

Notification of TOC limits exceed was made by cc:mail  
on 6/5/95 to John M. Conner, project coordinator. See comm  
Supporting data included in this data group. RWS 6-5-95  
6-6-95 RWS

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number,  
R = Replicate Number, A = Aliquot Code.

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT

TICTOC REV 2.0

<<< BLANK ANALYSIS >>>

Sample: BLK

Date: 06/03/95

Time: 17:13:24

Sample Size = 1 uL

Dil Factor = 1

Blank ID # = BLK

Blank Value = N/A

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	0.10	0.00
2	1.01	0.20	50.00
3	1.51	0.40	50.00
4	2.01	0.70	42.86
5	2.51	0.90	22.22
6	3.00	1.10	18.18
7	3.50	1.10	0.00
8	4.00	1.30	15.38
9	4.50	1.40	7.14
10	5.00	1.50	6.67
11	5.50	1.60	6.25
12	6.00	1.70	5.88
13	6.50	1.80	5.56
14	7.00	1.90	5.26
15	7.50	2.00	5.00
16	8.00	2.10	4.76
17	8.50	2.10	0.00
18	9.00	2.20	4.55
19	9.50	2.30	4.35
20	10.00	2.40	4.17
21	10.50	2.50	4.00
22	11.00	2.60	3.85

BLANK VALUE = 2.6 micrograms carbon

BLANK FACTOR = 2.6 / 10.99963 =

+2.4E-01 ug/min Carbon

Sample Run By:

KR MONTEITH

00000

SIGNATURE ABOVE REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT  
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 49 TO 68.

TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT

TICTOC REV 2.0

<<< BLANK ANALYSIS >>>

Sample: BLK

Date: 06/03/95

Time: 17:25:21

Sample Size = 1 uL

Dil Factor = 1

Blank ID # = BLK

Blank Value = N/A

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	== Analysis Time ==	== Coulometer ==	== % Difference ==
1	0.51	0.00	0.00
2	1.01	4.40	100.00
3	1.50	13.20	66.67
4	2.00	18.90	30.16
5	2.50	22.30	15.25
6	3.00	23.80	6.30
7	3.50	25.00	4.80
8	4.00	26.00	3.85
9	4.50	27.30	4.76
10	5.00	28.80	5.21
11	5.50	29.50	2.37
12	6.00	30.40	2.96
13	6.50	31.30	2.88
14	7.00	31.90	1.88
15	7.50	32.70	2.45
16	8.00	33.20	1.51
17	8.50	33.70	1.48
18	9.00	34.20	1.46
19	9.50	34.60	1.16
20	10.00	35.00	1.14
21	10.50	35.40	1.13
22	11.00	35.70	0.84

BLANK VALUE = 35.7 micrograms carbon

BLANK FACTOR = 35.7 / 10.99976 = +3.25E+00 ug/min Carbon

<<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!>>>>

Sample Run By:

KR MONTEITH

00000

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: STD

Date: 06/03/95

Time: 17:38:37

Sample Size = 750 uL

Dil Factor = 1

Blank ID # =

Blank Value = .24 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading	==== Analysis Time	==== Coulometer	==== % Difference ==
1	0.51	0.10	0.00
2	1.01	63.60	99.84
3	1.51	189.70	66.47
4	2.01	292.90	35.23
5	2.51	350.70	16.48
6	3.01	378.80	7.42
7	3.51	390.20	2.92
8	4.01	395.50	1.34
9	4.50	398.40	0.73
10	5.00	399.80	0.35
11	5.51	400.90	0.27
12	6.01	401.80	0.22
13	6.50	402.50	0.17
14	7.00	403.00	0.12
15	7.50	403.50	0.12
16	8.00	404.30	0.20
17	8.50	405.40	0.27
18	9.00	406.10	0.17
19	9.50	406.70	0.15
20	10.00	407.00	0.07
21	10.50	407.50	0.12
22	11.00	407.80	0.07

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 407.8 - 2.640176 ) (1) / (750) = +5.402E-01 g/L Carbon  
( 407.8 - 2.640176 ) (1) / (750) (12) = +4.502E-02 Molar Carbon

Sample Run By:

KR MONTEITH

00000

TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: STD

Date: 06/03/95

Time: 17:50:26

Sample Size = 250 uL

Dil Factor = 1

Blank ID # =

Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading	==== Analysis Time	==== Coulometer	==== % Difference ==
1	0.51	0.00	0.00
2	1.01	38.00	100.00
3	1.51	279.20	86.39
4	2.00	563.90	50.49
5	2.50	656.00	14.04
6	3.00	683.70	4.05
7	3.50	693.00	1.34
8	4.00	700.90	1.13
9	4.50	709.50	1.21
10	5.00	714.20	0.66
11	5.50	717.60	0.47
12	6.00	719.20	0.22
13	6.50	721.10	0.26
14	7.00	722.20	0.15
15	7.50	723.40	0.17
16	8.00	724.50	0.15
17	8.50	725.70	0.17
18	9.00	726.60	0.12
19	9.50	727.60	0.14
20	10.00	728.40	0.11
21	10.50	729.20	0.11
22	11.00	730.10	0.12

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 730.1 - 35.74643 ) (1)/(250) = +2.777E+00 g/L Carbon  
 ( 730.1 - 35.74643 ) (1)/(250) (12) = +2.315E-01 Molar Carbon  
 <<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!>>>>

Sample Run By:

KR MONTEITH

00000

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961

Date: 06/03/95

Time: 18:02:14

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = .24 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	0.50	0.00
2	1.01	7.40	93.24
3	1.51	35.90	79.39
4	2.00	107.40	66.57
5	2.50	218.90	50.94
6	3.00	328.40	33.34
7	3.50	400.00	17.90
8	4.00	436.40	8.34
9	4.50	451.20	3.28
10	5.00	458.80	1.66
11	5.50	462.10	0.71
12	6.00	464.80	0.58
13	6.50	466.30	0.32
14	7.00	467.70	0.30
15	7.50	468.80	0.23
16	8.00	469.90	0.23
17	8.50	471.00	0.23
18	9.00	471.80	0.17
19	9.50	472.90	0.23
20	10.00	473.50	0.13
21	10.50	474.00	0.11
22	11.00	474.60	0.13

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 474.6 - 2.640176 ) (1)/(1) = +4.720E+02 g/L Carbon  
( 474.6 - 2.640176 ) (1)/(1) (12) = +3.933E+01 Molar Carbon

Sample Run By:

KR MONTEITH

00000

\*  
SUPPORTING  
DATA

.0660g = 7.15E<sup>3</sup>

53

TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961

Date: 06/03/95

Time: 18:14:38

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	0.40	0.00
2	1.01	20.60	98.06
3	1.50	203.70	89.89
4	2.00	631.60	67.75
5	2.50	1173.70	46.19
6	3.00	1732.80	32.27
7	3.50	2226.70	22.18
8	4.00	2652.30	16.05
9	4.50	3064.30	13.45
10	5.00	3434.00	10.77
11	5.50	3748.90	8.40
12	6.00	3998.20	6.24
13	6.50	4185.10	4.47
14	7.00	4320.60	3.14
15	7.50	4413.20	2.10
16	8.00	4477.30	1.43
17	8.50	4522.80	1.01
18	9.00	4552.90	0.66
19	9.50	4573.70	0.45
20	10.00	4589.00	0.33
21	10.50	4598.20	0.20
22	11.00	4605.50	0.16

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 4605.5 - 35.74603 ) (1)/(1) = +4.5698E+03 g/L Carbon  
 ( 4605.5 - 35.74603 ) (1)/(1) (12) = +3.8081E+02 Molar Carbon  
 <<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!>>>>

Sample Run By:

KR MONTEITH

00000

\* SUPPORTIVE  
DATA

06609 = 6.92E4

S4

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961

Date: 06/03/95

Time: 18:28:14

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = .24 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	2.70	0.00
2	1.01	9.90	72.73
3	1.51	25.60	61.33
4	2.00	47.20	45.76
5	2.50	65.20	27.61
6	3.00	77.30	15.65
7	3.50	84.70	8.74
8	4.00	88.60	4.40
9	4.50	91.60	3.28
10	5.00	94.10	2.66
11	5.50	95.80	1.77
12	6.00	97.40	1.64
13	6.50	98.70	1.32
14	7.00	99.80	1.10
15	7.50	100.90	1.09
16	8.00	101.90	0.98
17	8.50	102.70	0.78
18	9.00	103.60	0.87
19	9.50	104.10	0.48
20	10.00	104.80	0.67
21	10.50	105.30	0.47
22	11.00	105.80	0.47

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 105.8 - 2.640176 ) (1)/(1) = +1.032E+02 g/L Carbon  
( 105.8 - 2.640176 ) (1)/(1) (12) = +8.597E+00 Molar Carbon

Sample Run By:

KR MONTEITH

00000

\* SUPPORT DATA

- 0.111g = 9.30E<sup>3</sup>

55



WHC-SD-WM-DP- 115, REV. 1  
 TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
 TIC TOC REV 2.0

Sample: 961

Date: 06/03/95

Time: 18:40:33

Sample Size = 1 uL  
 Dil Factor = 1  
 Blank ID # =  
 Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH  
 Min Readings = 22  
 Max Readings = 22  
 % Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	0.00	0.00
2	1.01	20.10	100.00
3	1.51	116.10	82.69
4	2.01	308.10	62.32
5	2.50	491.60	37.33
6	3.00	631.40	22.14
7	3.50	739.10	14.57
8	4.00	823.60	10.26
9	4.50	893.80	7.85
10	5.00	958.20	6.72
11	5.50	1011.50	5.27
12	6.00	1055.20	4.14
13	6.50	1093.90	3.54
14	7.00	1125.30	2.79
15	7.50	1152.60	2.37
16	8.00	1179.30	2.26
17	8.50	1199.90	1.72
18	9.00	1216.80	1.39
19	9.50	1229.70	1.05
20	10.00	1239.80	0.81
21	10.50	1247.60	0.63
22	11.00	1253.10	0.44

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 1253.1 - 35.75198 ) (1)/(1) = +1.2173E+03 g/L Carbon  
 ( 1253.1 - 35.75198 ) (1)/(1)(12) = +1.0145E+02 Molar Carbon  
 <<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!!!>>>>

Sample Run By:

KR MONTEITH

00000

\* SUPPORTING DATA

0.1118 = 1.10E<sup>-5</sup>

56

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961

Date: 06/03/95

Time: 18:53:05

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = .24 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	== Analysis Time ==	== Coulometer ==	== % Difference ==
1	0.51	5.50	0.00
2	1.01	21.40	74.30
3	1.50	33.20	35.54
4	2.00	48.70	31.83
5	2.50	62.60	22.20
6	3.00	74.00	15.41
7	3.50	80.70	8.30
8	4.00	84.20	4.16
9	4.50	86.40	2.55
10	5.00	87.50	1.26
11	5.50	88.50	1.13
12	6.00	89.20	0.78
13	6.50	89.90	0.78
14	7.00	90.60	0.77
15	7.50	91.10	0.55
16	8.00	91.60	0.55
17	8.50	92.10	0.54
18	9.00	92.50	0.43
19	9.50	93.10	0.64
20	10.00	93.50	0.43
21	10.50	93.80	0.32
22	11.00	94.30	0.53

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 94.3 - 2.639736 ) (1)/(1) = +9.17E+01 g/L Carbon  
( 94.3 - 2.639736 ) (1)/(1) (12) = +7.64E+00 Molar Carbon

Sample Run By:

KR MONTEITH

00000

\* 0098g = 9.36E3

52

TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961

Date: 06/03/95

Time: 20:00:37

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	0.10	0.00
2	1.01	17.30	99.42
3	1.50	107.10	83.85
4	2.00	300.20	64.32
5	2.50	504.60	40.51
6	3.00	667.80	24.44
7	3.50	786.90	15.14
8	4.00	883.30	10.91
9	4.50	968.30	8.78
10	5.00	1045.00	7.34
11	5.50	1117.80	6.51
12	6.00	1182.10	5.44
13	6.50	1237.50	4.48
14	7.00	1287.70	3.90
15	7.50	1330.80	3.24
16	8.00	1368.20	2.73
17	8.50	1398.50	2.17
18	9.00	1423.60	1.76
19	9.50	1443.70	1.39
20	10.00	1460.50	1.15
21	10.50	1472.90	0.84
22	11.00	1482.40	0.64

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 1482.4 - 35.74603 ) (1)/(1) = +1.4467E+03 g/L Carbon  
 ( 1482.4 - 35.74603 ) (1)/(1) (12) = +1.2055E+02 Molar Carbon  
 <<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!!!>>>>

Sample Run By:

KR MONTEITH

00000

\*.0098g = 1.48e<sup>-5</sup>

WHC-SD-WM-DP- 115, REV. 2  
TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961 DJF Date: 06/03/95 Time: 20:16:28

Sample Size = 1 uL Analyst : KR MONTEITH  
Dil Factor = 1 Min Readings = 22  
Blank ID # = Max Readings = 22  
Blank Value = .24 ug/minute C % Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	0.00	0.00
2	1.01	8.80	100.00
3	1.51	17.30	49.13
4	2.00	29.20	40.75
5	2.50	43.40	32.72
6	3.00	53.50	18.88
7	3.50	58.30	8.23
8	4.00	61.20	4.74
9	4.50	62.50	2.08
10	5.00	63.40	1.42
11	5.50	64.30	1.40
12	6.00	65.00	1.08
13	6.50	65.50	0.76
14	7.00	66.00	0.76
15	7.50	66.50	0.75
16	8.00	67.10	0.89
17	8.50	67.30	0.30
18	9.00	67.70	0.59
19	9.50	68.10	0.59
20	10.00	68.50	0.58
21	10.50	68.90	0.58
22	11.00	69.30	0.58

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 69.3 - 2.639472 ) (1)/(1) = +6.67E+01 g/L Carbon  
( 69.3 - 2.639472 ) (1)/(1) (12) = +5.56E+00 Molar Carbon

Sample Run By: KR MONTEITH 00000

\*.0082g = 8.13e<sup>3</sup>

59

TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961 D5Q

Date: 06/03/95

Time: 20:34:28

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading	==== Analysis Time	==== Coulometer	==== % Difference ==
1	0.51	0.10	0.00
2	1.01	30.00	99.67
3	1.51	156.60	80.84
4	2.00	347.60	54.95
5	2.50	505.60	31.25
6	3.00	622.90	18.83
7	3.50	709.70	12.23
8	4.00	781.50	9.19
9	4.50	838.30	6.78
10	5.00	886.20	5.41
11	5.50	925.30	4.23
12	6.00	957.90	3.40
13	6.50	985.90	2.84
14	7.00	1009.60	2.35
15	7.50	1030.20	2.00
16	8.00	1048.20	1.72
17	8.50	1062.50	1.35
18	9.00	1074.20	1.09
19	9.50	1083.40	0.85
20	10.00	1091.20	0.71
21	10.50	1096.80	0.51
22	11.00	1101.50	0.43

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 1101.5 - 35.7496 ) (1)/(1) = +1.0658E+03 g/L Carbon  
 ( 1101.5 - 35.7496 ) (1)/(1) (12) = +8.8813E+01 Molar Carbon  
 <<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!!!>>>>

Sample Run By:

KR MONTEITH

00000

\* .0082g = 1.30e5

4.0

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961 + SPK

Date: 06/03/95

Time: 20:47:28

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = .24 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	== Analysis Time ==	== Coulometer ==	== % Difference ==
1	0.51	0.60	0.00
2	1.01	17.80	96.63
3	1.51	50.30	64.61
4	2.00	81.10	37.98
5	2.50	103.70	21.79
6	3.00	116.70	11.14
7	3.50	122.10	4.42
8	4.00	125.10	2.40
9	4.50	126.70	1.26
10	5.00	127.90	0.94
11	5.50	128.90	0.78
12	6.00	129.60	0.54
13	6.50	130.30	0.54
14	7.00	131.00	0.53
15	7.50	131.50	0.38
16	8.00	132.10	0.45
17	8.50	132.60	0.38
18	9.00	133.20	0.45
19	9.50	133.60	0.30
20	10.00	134.10	0.37
21	10.50	134.60	0.37
22	11.00	135.10	0.37

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 135.1 - 2.639971 ) (1)/(1) =

+1.325E+02 g/L Carbon

( 135.1 - 2.639971 ) (1)/(1) (12) =

+1.104E+01 Molar Carbon

Sample Run By:

KR MONTEITH

00000

$$\frac{(135.1 - 2.64) - [(94.3 - 2.64)(0.93878)]}{61 \times 600 (1.1)}$$

\*.0092g + .1mc 15N12F

= 77.3%

TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 961 + SPK

Date: 06/03/95

Time: 20:59:21

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	== Analysis Time ==	== Coulometer ==	== % Difference ==
1	0.51	0.00	0.00
2	1.01	53.20	100.00
3	1.50	311.10	82.90
4	2.00	635.10	51.02
5	2.50	875.30	27.44
6	3.00	1037.60	15.64
7	3.50	1156.00	10.24
8	4.00	1254.10	7.82
9	4.50	1340.40	6.44
10	5.00	1414.30	5.23
11	5.50	1477.40	4.27
12	6.00	1528.60	3.35
13	6.50	1569.80	2.62
14	7.00	1605.50	2.22
15	7.50	1636.00	1.86
16	8.00	1662.80	1.61
17	8.50	1685.80	1.36
18	9.00	1704.50	1.10
19	9.50	1719.60	0.88
20	10.00	1732.30	0.73
21	10.50	1742.30	0.57
22	11.00	1750.50	0.47

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 1750.5 - 35.74921 ) (1)/(1) = +1.7148E+03 g/L Carbon  
 ( 1750.5 - 35.74921 ) (1)/(1) (12) = +1.4290E+02 Molar Carbon  
 <<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!>>>>

Sample Run By:

KR MONTEITH

00000

$$* \frac{(1750.5 - 35.7) \left[ \frac{(1402.4 - 35.7)(.93878)}{2998(.1)} \right]}{.00922 + .1 \text{ mc } 16N12} \times 100 = 118.9$$

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 963

Date: 06/03/95

Time: 21:11:19

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = .24 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading	==== Analysis Time	==== Coulometer	==== % Difference ==
1	0.51	5.90	0.00
2	1.01	15.60	62.18
3	1.51	47.60	67.23
4	2.00	87.20	45.41
5	2.50	115.10	24.24
6	3.00	129.60	11.19
7	3.50	135.80	4.57
8	4.00	138.80	2.16
9	4.50	140.60	1.28
10	5.00	142.10	1.06
11	5.50	143.10	0.70
12	6.00	144.00	0.62
13	6.50	144.80	0.55
14	7.00	145.60	0.55
15	7.50	146.40	0.55
16	8.00	147.10	0.48
17	8.50	147.70	0.41
18	9.00	148.40	0.47
19	9.50	148.90	0.34
20	10.00	149.50	0.40
21	10.50	150.10	0.40
22	11.00	150.60	0.33

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 150.6 - 2.639033 ) (1)/(1) = +1.480E+02 g/L Carbon  
( 150.6 - 2.639033 ) (1)/(1) (12) = +1.233E+01 Molar Carbon

Sample Run By:

KR MONTEITH

00000

\*.01072 = 1.38E<sup>-4</sup>

63



TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 963

Date: 06/03/95

Time: 21:24:25

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading	==== Analysis Time	==== Coulometer	==== % Difference ==
1	0.51	0.00	0.00
2	1.01	12.30	100.00
3	1.50	100.00	87.70
4	2.00	270.20	62.99
5	2.50	410.50	34.18
6	3.00	509.90	19.49
7	3.50	588.70	13.39
8	4.00	656.10	10.27
9	4.50	715.30	8.28
10	5.00	768.80	6.96
11	5.50	816.20	5.81
12	6.00	854.20	4.45
13	6.50	888.50	3.86
14	7.00	918.60	3.28
15	7.50	943.40	2.63
16	8.00	963.90	2.13
17	8.50	980.10	1.65
18	9.00	992.80	1.28
19	9.50	1002.00	0.92
20	10.00	1008.80	0.67
21	10.50	1014.10	0.52
22	11.00	1017.80	0.36

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 1017.8 - 35.75198 ) (1)/(1) = +9.8205E+02 g/L Carbon  
 ( 1017.8 - 35.75198 ) (1)/(1) (12) = +8.1837E+01 Molar Carbon  
 <<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!>>>>

Sample Run By:

KR MONTEITH

00000

\*.01072 = 9.18E-4

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 963

Date: 06/03/95

Time: 21:38:42

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = .24 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	0.60	0.00
2	1.01	10.40	94.23
3	1.50	33.90	69.32
4	2.00	58.10	41.65
5	2.50	76.00	23.55
6	3.00	86.90	12.54
7	3.50	92.80	6.36
8	4.00	95.80	3.13
9	4.50	97.80	2.04
10	5.00	98.90	1.11
11	5.50	100.00	1.10
12	6.00	100.90	0.89
13	6.50	101.60	0.69
14	7.00	102.30	0.68
15	7.50	102.80	0.49
16	8.00	103.50	0.68
17	8.50	104.10	0.58
18	9.00	104.60	0.48
19	9.50	105.20	0.57
20	10.00	105.70	0.47
21	10.50	106.20	0.47
22	11.00	106.70	0.47

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 106.7 - 2.639707 ) (1)/(1) = +1.041E+02 g/L Carbon  
( 106.7 - 2.639707 ) (1)/(1) (12) = +8.672E+00 Molar Carbon

Sample Run By:

KR MONTEITH

00000

*Support Data  
RWS  
6-5-95\**

*.0097g = 1.07%*

65

TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 963

Date: 06/03/95

Time: 21:50:45

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading	==== Analysis Time	==== Coulometer	==== % Difference ==
1	0.51	0.00	0.00
2	1.01	9.30	100.00
3	1.51	65.90	85.89
4	2.00	207.40	68.23
5	2.50	394.40	47.41
6	3.00	554.80	28.91
7	3.50	681.30	18.57
8	4.00	780.20	12.68
9	4.50	862.50	9.54
10	5.00	933.80	7.64
11	5.50	1002.60	6.86
12	6.00	1068.10	6.13
13	6.50	1127.90	5.30
14	7.00	1182.80	4.64
15	7.50	1230.30	3.86
16	8.00	1273.90	3.42
17	8.50	1312.40	2.93
18	9.00	1345.60	2.47
19	9.50	1374.30	2.09
20	10.00	1398.80	1.75
21	10.50	1419.90	1.49
22	11.00	1437.20	1.20

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 1437.2 - 35.74365 ) (1)/(1) = +1.4015E+03 g/L Carbon  
 ( 1437.2 - 35.74365 ) (1)/(1) (12) = +1.1679E+02 Molar Carbon  
 <<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!>>>>

*SUPPORTING DATA*  
*6-5-95*

Sample Run By:

KR MONTEITH

00000

.0097g = 1.44E 5

TIC- TOTAL INORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 963

Date: 06/03/95

Time: 22:03:31

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = .24 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	==== Analysis Time ==	==== Coulometer ==	==== % Difference ==
1	0.51	14.60	0.00
2	1.01	26.00	43.85
3	1.51	46.20	43.72
4	2.01	69.30	33.33
5	2.51	86.60	19.98
6	3.00	96.50	10.26
7	3.50	101.00	4.46
8	4.00	103.70	2.60
9	4.50	105.70	1.89
10	5.00	107.10	1.31
11	5.50	108.20	1.02
12	6.00	109.30	1.01
13	6.50	110.20	0.82
14	7.00	111.10	0.81
15	7.50	111.80	0.63
16	8.00	112.70	0.80
17	8.50	113.40	0.62
18	9.00	114.10	0.61
19	9.50	114.60	0.44
20	10.00	115.30	0.61
21	10.50	115.90	0.52
22	11.00	116.50	0.52

USER INPUT BLANK VALUE

BLANK VALUE = 2.639941 micrograms carbon

BLANK FACTOR = 2.639941 / 10.99976 = +2.4E-01 ug/min Carbon

SAMPLE RESULTS:

( 116.5 - 2.639971 ) (1)/(1) = +1.139E+02 g/L Carbon  
( 116.5 - 2.639971 ) (1)/(1) (12) = +9.488E+00 Molar Carbon

Sample Run By:

KR MONTEITH

00000

Supporting  
\* data

.0094g = 1.21E-4

67

WHC-SD-WM-DP- 115, REV. 1  
TOC- TOTAL ORGANIC CARBON ANALYSIS REPORT  
TICTOC REV 2.0

Sample: 963

Date: 06/03/95

Time: 22:16:34

Sample Size = 1 uL

Dil Factor = 1

Blank ID # =

Blank Value = 3.25 ug/minute C

Analyst : KR MONTEITH

Min Readings = 22

Max Readings = 22

% Difference = 10

== Reading ==	== Analysis Time ==	== Coulometer ==	== % Difference ==
1	0.51	0.30	0.00
2	1.01	16.00	98.13
3	1.51	86.20	81.44
4	2.00	224.80	61.65
5	2.50	348.40	35.48
6	3.00	441.60	21.11
7	3.50	520.10	15.09
8	4.00	590.90	11.98
9	4.50	653.40	9.57
10	5.00	712.80	8.33
11	5.50	766.80	7.04
12	6.00	814.10	5.81
13	6.50	854.90	4.77
14	7.00	890.20	3.97
15	7.50	920.30	3.27
16	8.00	945.00	2.61
17	8.50	964.30	2.00
18	9.00	978.20	1.42
19	9.50	988.20	1.01
20	10.00	995.00	0.68
21	10.50	999.80	0.48
22	11.00	1003.40	0.36

USER INPUT BLANK VALUE

BLANK VALUE = 35.74921 micrograms carbon

BLANK FACTOR = 35.74921 / 10.99976 = +3.3E+00 ug/min Carbon

SAMPLE RESULTS:

( 1003.4 - 35.74326 ) (1)/(1) = +9.6766E+02 g/L Carbon  
( 1003.4 - 35.74326 ) (1)/(1) (12) = +8.0638E+01 Molar Carbon  
<<<< WARNING - BLANK VALUE EXCEEDS 1.5 ug/min Carbon!!!!>>>>

Sample Run By:

KR MONTEITH

00000

*Supporting  
\* data*

*.0094g = 1.03E<sup>-5</sup>*

WHC-SD-WM-DP-115, REV. 1

## RADIOCHEMICAL ANALYSES

**WHC-SD-WM-DP-115, REV. 1**

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# LABCORE Data Entry Template for Worklist# 1397

Analyst: ZNT Instrument: AB00 16 Book # 107B52  
0-495 LAB  
Method: LA-508-101 Rev/Mod 4-2 D-2 WB 27806

Worklist Comment: If dose rate warrents, run under RWP 273. SLF

GROUP	PROJECT	S	TYPE	SAMPLE#	R	A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1	STD				@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
		1	STD				@ALPHA01 ALPHA01E	SOLID			N/A	% Ct. Error
		2	BLNK-PREP				@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
		2	BLNK-PREP				@ALPHA01 ALPHA01E	SOLID			N/A	% Ct. Error
		3	BLNK/BKG	<u>1, c</u>			@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
95000069	C-204	4	SAMPLE	S95T000879	0	F	@ALPHA01 ALPHA01	SOLID	N/A			uCi/g
95000069	C-204	4	SAMPLE	S95T000879	0	F	@ALPHA01 ALPHA01E	SOLID	N/A			% Ct. Error
95000069	C-204	5	DUP	S95T000879	0	F	@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
95000069	C-204	5	DUP	S95T000879	0	F	@ALPHA01 ALPHA01E	SOLID			N/A	% Ct. Error
95000069	C-204	6	SPK	S95T000879	0	F	@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
95000069	C-204	7	SAMPLE	S95T000882	0	F	@ALPHA01 ALPHA01	SOLID	N/A			uCi/g
95000069	C-204	7	SAMPLE	S95T000882	0	F	@ALPHA01 ALPHA01E	SOLID	N/A			% Ct. Error
95000069	C-204	8	DUP	S95T000882	0	F	@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
95000069	C-204	8	DUP	S95T000882	0	F	@ALPHA01 ALPHA01E	SOLID			N/A	% Ct. Error

Final page for worklist # 1397

Sharon Mif 5-16-95  
Analyst Signature Date

Sharon L. Uddell 05-17-95  
Analyst Signature Date

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.



PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP- 115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3) LIQUIDS

		STANDARD	REPLICATE
Type	DETECTOR NUMBER	16	16
STANDARD	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	3810	3730
1397	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.4	0.4
AT	SAMPLE SIZE in mL (SS)	10.000	10.000
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST DILUTION FACTOR (DDF)	1	1
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
LIQUID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	126.600	123.933
Sample #	Sample Concentration in $\mu\text{Ci/L}$	2.71E-02	BOOK#
WORKLIST#1397	Replicate Concentration in $\mu\text{Ci/L}$	2.65E-02	107B52
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/L}$	2.6819E-02	
Analyst			
TLM	Rs (Sample Count Rate) = (TC / CT) - BKG		
Date	ALPHA TOTAL $\mu\text{Ci/L}$ = Rs * 1000mL/L * DF * DDF / ( EFF * SS * 2220000dpm/ $\mu\text{Ci}$ )		
05/16/95	ALPHA TOTAL $\mu\text{Ci/mL}$ = ALPHA TOTAL $\mu\text{Ci/L}$ / 1000mL/L		
Time	Relative Counting Error = [  (The Square Root of TC + BKG * CT) / (TC - BKG * CT)  ] * 1.96 * 100		
10:30 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

v RESULTS v

ALPHA TOTAL in $\mu\text{Ci/mL}$ (Average)	=	2.68E-05	DETECTION LEVEL
			1.35E-07 $\mu\text{Ci/mL}$
RELATIVE COUNTING ERROR	=	3.2%	

Data Entry by: *Sharon L. Gifford* Date: 05/17/95  
 Approved by: *Jeff* Date: 5/18/95

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP-115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3)

SOLIDS

		BLANK	REPLICATE
Type	DETECTOR NUMBER	16	16
BLANK	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	12	12
1397	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.4	0.4
AT	SAMPLE SIZE in mL (SS)	0.100	0.100
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	2.0536	2.0536
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
SOLID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	0.269	0.269
Sample #	Sample Concentration in $\mu\text{Ci/g}$	< 2.81E-03	BOOK#
S95T879	Replicate Concentration in $\mu\text{Ci/g}$	< 2.81E-03	
Instrument Code			
WB27806	Maximum Concentration in $\mu\text{Ci/g}$	< 2.8090E-03	
Analyst			
TLM	Rs (Sample Count Rate) = (TC / CT) - BKG		
Date	ALPHA TOTAL $\mu\text{Ci/g}$ = Rs * 1000mL/L * DF / ( EFF * SS * Dg/L * 2220000dpm/ $\mu\text{Ci}$ )		
05/16/95			
Time	Relative Counting Error = [  (The Square Root of TC + BKG * CT) / (TC - BKG * CT)  ] * 1.96 * 100		
10:30 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

RESULTS

ALPHA TOTAL	in $\mu\text{Ci/g}$	(Maximum)	=	< 2.81E-03	DETECTION LEVEL
LESS Than Value was Determined from Lc.					
RELATIVE COUNTING ERROR			=	500.0%	6.56E-03 $\mu\text{Ci/g}$

Data Entry by: <i>Sharon D. Hest</i>	Date: 05/17/95
Approved by: <i>[Signature]</i>	Date: 5/18/95

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP-115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3)

SOLIDS

		SAMPLE	REPLICATE
Type	DETECTOR NUMBER	16	16
SAMPLE	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	32	29
1397	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.4	0.4
AT	SAMPLE SIZE in mL (SS)	0.100	0.100
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	2.0536	2.0536
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
SOLID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	0.667	0.567
Sample #	Sample Concentration in $\mu\text{Ci/g}$	6.95E-03	BOOK#
S95T879	Replicate Concentration in $\mu\text{Ci/g}$	5.91E-03	
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/g}$	6.4289E-03	
Analyst			
TLM	Rs (Sample Count Rate) = (TC / CT) - BKG		
Date	ALPHA TOTAL $\mu\text{Ci/g}$ = Rs * 1000mL/L * DF / ( EFF * SS * Dg/L * 2220000dpm/ $\mu\text{Ci}$ )		
05/16/95			
Time	Relative Counting Error = [  (The Square Root of TC + BKG * CT) / (TC - BKG * CT)  ] * 1.96 * 100		
10:30 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

RESULTS

ALPHA TOTAL	in $\mu\text{Ci/g}$ (Average)	=	6.43E-03	DETECTION LEVEL
				6.56E-03 $\mu\text{Ci/g}$
RELATIVE COUNTING ERROR		=	73.8%	

Data Entry by: <i>Sharon L. H. G. G.</i>	Date: 05/17/95
Approved by: <i>[Signature]</i>	Date: 5/17/95

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP-115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3)

SOLIDS

		SAMPLE	REPLICATE
Type	DETECTOR NUMBER	18	18
DUPLICATE	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	49	61
1397	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.4	0.4
AT	SAMPLE SIZE in mL (SS)	0.100	0.100
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	2.1156	2.1156
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
SOLID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	1.233	1.633
Sample #	Sample Concentration in $\mu\text{Ci/g}$	1.25E-02	BOOK#
S95T879	Replicate Concentration in $\mu\text{Ci/g}$	1.65E-02	
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/g}$	1.4505E-02	
Analyst			
TLM	Rs (Sample Count Rate) = (TC / CT) - BKG		
Date	ALPHA TOTAL $\mu\text{Ci/g}$ = Rs * 1000mL/L * DF / ( EFF * SS * Dg/L * 2220000dpm/ $\mu\text{Ci}$ )		
05/16/95			
Time	Relative Counting Error = [  (The Square Root of TC + BKG * CT) / (TC - BKG * CT)  ] * 1.96 * 100		
10:30 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

RESULTS

ALPHA TOTAL in $\mu\text{Ci/g}$ (Average)	=	1.45E-02	DETECTION LEVEL
			6.37E-03 $\mu\text{Ci/g}$
RELATIVE COUNTING ERROR	=	41.4%	

Data Entry by: <i>Sharon L. H. H.</i>	Date: 05/17/95
Approved by: <i>[Signature]</i>	Date: 5/17/95

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP- 115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3) SOLIDS

Type	DETECTOR NUMBER	SPIKE	REPLICATE
SPIKE	DISH SIZE 1, 2, or 5 (MS)	16	16
Work List	TOTAL COUNTS (TC)	2	2
1397	COUNT TIME in MINUTES (CT)	31673	31450
AT or TB ?	BACKGROUND in cpm (BKG)	30	30
AT	SAMPLE VOLUME in mL (Spiked Vial) (SS)	0.4	0.4
Test Code	SAMPLE DILUTION FACTOR (Spiked Vial) (DF)	0.100	0.100
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	1	1
Matrix	SPIKE VOLUME in mL (SVol)	2.0536	2.0536
SOLID	SPIKE DILUTION FACTOR (SDF)	0.100	0.100
Sample #	SPIKE VALUE in $\mu\text{Ci/L}$ (SVal)	1	1
S95T879	INSTRUMENT EFFICIENCY FACTOR (EFF)	36.372	36.372
Instrument Code	SAMPLE + SPIKE $\mu\text{Ci/g}$ (S+S)	0.2104	0.2104
WB27806	AVERAGE or MAXIMUM $\mu\text{Ci/g}$ from FORM C	1.10E+01	1.09E+01
Analyst	BOOK#	6.4289E-03	
TLM		94B43	
Date	Rs (Sample Count Rate) = (TC / CT) - BKG		
05/16/95	SAMPLE + SPIKE $\mu\text{Ci/g}$ = Rs * 1000mL/L * DF / ( EFF * SS * Dg/L * 2220000dpm/ $\mu\text{Ci}$ )		
Time	PERCENT SPIKE RECOVERY = (((S+S $\mu\text{Ci/g}$ - SAMPLE $\mu\text{Ci/g}$ ) * ((SDF/SVol)/(DF/SS/Dg/L)))/(SVal)*100		
10:30 AM			

RESULT **AVG. PERCENT SPIKE RECOVERY** = **61.9%**

Data Entry by: Sharon L. G. L. Date: 17-May-95  
 Approved by: St. R. H. Date: 5/17/95  
 Form 508101\_X Rev. 1.3 Page 1 of 1

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP-115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3)

SOLIDS

		SAMPLE	REPLICATE
Type	DETECTOR NUMBER	16	16
SAMPLE	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	63	94
1397	COUNT TIME in MINUTES (CT)	30	30
AT or IB ?	BACKGROUND in cpm (BKG)	0.4	0.4
AT	SAMPLE SIZE in mL (SS)	0.100	0.100
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	2.0304	2.0304
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
SOLID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	1.700	2.733
Sample #	Sample Concentration in $\mu\text{Ci/g}$	1.79E-02	BOOK#
S95T882	Replicate Concentration in $\mu\text{Ci/g}$	2.88E-02	
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/g}$	2.3373E-02	
Analyst			
TLM	Rs (Sample Count Rate) = $(TC / CT) - BKG$		
Date	ALPHA TOTAL $\mu\text{Ci/g}$ = $Rs * 1000\text{mL/L} * DF / (EFF * SS * Dg/L * 2220000\text{dpm}/\mu\text{Ci})$		
05/16/95			
Time	Relative Counting Error = $[ (The Square Root of TC + BKG * CT) / (TC - BKG * CT) ] * 1.96 * 100$		
10:30 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

RESULTS

ALPHA TOTAL	in $\mu\text{Ci/g}$	(Average)	=	2.34E-02	DETECTION LEVEL
					6.64E-03
RELATIVE COUNTING ERROR					$\mu\text{Ci/g}$
				=	33.3%

Data Entry by: *Shyan L. H. H.* Date: 05/17/95  
 Approved by: *Shyan L. H. H.* Date: 5/17/95  
 Form 508101\_C Rev. 1.3 Page 1 of 1

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP-//5, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3)

SOLIDS

		SAMPLE	REPLICATE
Type	DETECTOR NUMBER	16	16
DUPLICATE	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	48	48
1397	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.4	0.4
AT	SAMPLE SIZE in mL (SS)	0.100	0.100
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	2.0676	2.0676
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
SOLID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	1.200	1.133
Sample #	Sample Concentration in $\mu\text{Ci/g}$	1.24E-02	BOOK#
S95T882	Replicate Concentration in $\mu\text{Ci/g}$	1.17E-02	
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/g}$	1.2080E-02	
Analyst			
TLM	Rs (Sample Count Rate) = (TC / CT) - BKG		
Date	ALPHA TOTAL $\mu\text{Ci/g}$ = Rs * 1000mL/L * DF / ( EFF * SS * Dg/L * 2220000dpm/ $\mu\text{Ci}$ )		
05/16/95			
Time	Relative Counting Error = [  (The Square Root of TC + BKG * CT) / (TC - BKG * CT)  ] * 1.96 * 100		
10:30 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

▼ RESULTS ▼

ALPHA TOTAL in $\mu\text{Ci/g}$ (Average)	=	1.21E-02	DETECTION LEVEL
			6.52E-03 $\mu\text{Ci/g}$
RELATIVE COUNTING ERROR	=	43.9%	

Data Entry by: <i>Sharon L. Hyatt</i>	Date: 05/17/95
Approved by: <i>[Signature]</i>	Date: 5/17/95

# LABCORE Data Entry Template for Worklist# 1426

Analyst: LM Instrument: AB00 16 Book # 115352

Method: LA-508-101 Rev/Mod A-2 SLF 6-1-95 U327806

Worklist Comment: Determine sample size using Ludlum. SLF

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
		1 STD			@ALPHA01 ALPHA01E	SOLID			N/A	% Ct. Error
		2 BLNK-PREP			@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
		2 BLNK-PREP			@ALPHA01 ALPHA01E	SOLID			N/A	% Ct. Error
		3 BLNK/BKG	3.87		@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
95000069	C-204	4 SAMPLE	S95T000891	0 F	@ALPHA01 ALPHA01	SOLID	N/A			uCi/g
95000069	C-204	4 SAMPLE	S95T000891	0 F	@ALPHA01 ALPHA01E	SOLID	N/A			% Ct. Error
95000069	C-204	5 DUP	S95T000891	0 F	@ALPHA01 ALPHA01	SOLID			N/A	uCi/g
95000069	C-204	5 DUP	S95T000891	0 F	@ALPHA01 ALPHA01E	SOLID			N/A	% Ct. Error

Final page for worklist # 1426

Kevin M. 5/23/95  
Analyst Signature Date

Kevin M. 5/23/95  
Analyst Signature Date

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.



PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP- 115, REV 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3) LIQUIDS

		STANDARD	REPLICATE
Type	DETECTOR NUMBER	16	16
STANDARD	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	3742	4010
1426	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.5	0.5
AT	SAMPLE SIZE in mL (SS)	10.000	10.000
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST DILUTION FACTOR (DDF)	1	1
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
LIQUID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	124.233	133.167
Sample #	Sample Concentration in $\mu\text{Ci/L}$	2.66E-02	BOOK#
WORKLIST#1426	Replicate Concentration in $\mu\text{Ci/L}$	2.85E-02	115B52
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/L}$	2.7554E-02	
Analyst			
TLM	Rs (Sample Count Rate) = (TC / CT) - BKG		
Date	ALPHA TOTAL $\mu\text{Ci/L}$ = $R_s * 1000\text{mL/L} * DF * DDF / (EFF * SS * 2220000\text{dpm}/\mu\text{Ci})$		
05/23/95	ALPHA TOTAL $\mu\text{Ci/mL}$ = ALPHA TOTAL $\mu\text{Ci/L} / 1000\text{mL/L}$		
Time	Relative Counting Error = $[(\text{The Square Root of } TC + BKG * CT) / (TC - BKG * CT)] * 1.96 * 100$		
09:45 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

▼ RESULTS ▼

ALPHA TOTAL in $\mu\text{Ci/mL}$ (Average)	=	2.76E-05	DETECTION LEVEL
			1.48E-07 $\mu\text{Ci/mL}$
RELATIVE COUNTING ERROR	=	3.2%	

Data Entry by: Sharon G. G. G. Date: 05/24/95  
 Approved by: [Signature] Date: 5/24/95  
 Form 508101\_C Rev. 1.3 Page 1 of 1

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP-115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3)

SOLIDS

		BLANK	REPLICATE
Type	DETECTOR NUMBER	16	16
BLANK	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	67	49
1426	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.5	0.5
AT	SAMPLE SIZE in mL (SS)	0.100	0.100
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	2.2284	2.2284
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
SOLID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	1.733	1.133
Sample #	Sample Concentration in $\mu\text{Ci/g}$	1.67E-02	BOOK#
S95T891	Replicate Concentration in $\mu\text{Ci/g}$	1.09E-02	
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/g}$	1.3771E-02	
Analyst			
TLM	$R_s$ (Sample Count Rate) = $(TC / CT) - BKG$		
Date	$\text{ALPHA TOTAL } \mu\text{Ci/g} = R_s * 1000\text{mL/L} * DF / (EFF * SS * \text{Dg/L} * 2220000\text{dpm}/\mu\text{Ci})$		
05/23/95			
Time	Relative Counting Error = $[(\text{The Square Root of } TC + BKG * CT) / (TC - BKG * CT)] * 1.96 * 100$		
09:45 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

▼ RESULTS ▼

ALPHA TOTAL in $\mu\text{Ci/g}$ (Average)	=	1.38E-02	DETECTION LEVEL
			6.66E-03 $\mu\text{Ci/g}$
RELATIVE COUNTING ERROR	=	46.1%	

Data Entry by: <i>Sharon L. L...</i>	Date: 05/24/95
Approved by: <i>[Signature]</i>	Date: 5/24/95

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP- 115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3)

SOLIDS

		SAMPLE	REPLICATE
Type	DETECTOR NUMBER	16	16
SAMPLE	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	185	164
1426	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.5	0.5
AT	SAMPLE SIZE in mL (SS)	0.100	0.100
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	2.2284	2.2284
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
SOLID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	5.667	4.967
Sample #	Sample Concentration in $\mu\text{Ci/g}$	5.44E-02	BOOK#
S95T891	Replicate Concentration in $\mu\text{Ci/g}$	4.77E-02	
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/g}$	5.1080E-02	
Analyst			
TLM	$R_s$ (Sample Count Rate) = $(TC / CT) - BKG$		
Date	ALPHA TOTAL $\mu\text{Ci/g}$ = $R_s * 1000\text{mL/L} * DF / (EFF * SS * Dg/L * 2220000\text{dpm}/\mu\text{Ci})$		
05/23/95			
Time	Relative Counting Error = $[(\text{The Square Root of } TC + BKG * CT) / (TC - BKG * CT)] * 1.96 * 100$		
09:45 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

v RESULTS v

ALPHA TOTAL	in $\mu\text{Ci/g}$	(Average)	=	5.11E-02	DETECTION LEVEL
					6.66E-03
RELATIVE COUNTING ERROR			=	17.6%	$\mu\text{Ci/g}$

Data Entry by: <u>Sharon L. Lyle</u>	Date: 05/24/95
Approved by: <u>[Signature]</u>	Date: <u>5/24/95</u>

PLACE ANALYTICAL CARD IN BOX BELOW OR ATTACH TRAVELER

WHC-SD-WM-DP-115, REV. 1

AT : LA-508-101 (D-2)

LA-548-101 (A-3)

SOLIDS

		SAMPLE	REPLICATE
Type	DETECTOR NUMBER	16	16
DUPLICATE	DISH SIZE 1, 2, or 5 (MS)	2	2
Work List	TOTAL COUNTS (TC)	173	175
1426	COUNT TIME in MINUTES (CT)	30	30
AT or TB ?	BACKGROUND in cpm (BKG)	0.5	0.5
AT	SAMPLE SIZE in mL (SS)	0.100	0.100
Test Code	DILUTION FACTOR (DF)	1	1
@ALPHA01	DIGEST GRAMS of SOLIDS/L (Dg/L)	2.1852	2.1852
Matrix	EFFICIENCY FACTOR (EFF)	0.2104	0.2104
SOLID	Lc, Rmax, or Rs, (SAMPLE RATE) as APPROPRIATE	5.267	5.333
Sample #	Sample Concentration in $\mu\text{Ci/g}$	5.16E-02	BOOK#
S95T891	Replicate Concentration in $\mu\text{Ci/g}$	5.23E-02	
Instrument Code			
WB27806	Average Concentration in $\mu\text{Ci/g}$	5.1926E-02	
Analyst			
TLM	$R_s$ (Sample Count Rate) = $(TC / CT) - BKG$		
Date	$\text{ALPHA TOTAL } \mu\text{Ci/g} = R_s * 1000\text{mL/L} * DF / (EFF * SS * Dg/L * 2220000\text{dpm}/\mu\text{Ci})$		
05/23/95			
Time	Relative Counting Error = $[(\text{The Square Root of } TC + BKG * CT) / (TC - BKG * CT)] * 1.96 * 100$		
09:45 AM	Detection Levels and Less Than Values are determined from Procedure LA-508-002.		

RESULTS

ALPHA TOTAL in $\mu\text{Ci/g}$ (Average)	=	5.19E-02	DETECTION LEVEL
			6.79E-03 $\mu\text{Ci/g}$
RELATIVE COUNTING ERROR	=	17.0%	

Data Entry by: <i>[Signature]</i>	Date: 05/24/95
Approved by: <i>[Signature]</i>	Date: 5/24/95



Westinghouse  
Hanford Company

WHC-SD-WM-DP-115, REV. 1

P.O. Box 1970 Richland, WA 99352

PART II

WHC-SD-WM-DP-115, REV. 0-A

## ANALYTICAL SERVICES

90-DAY SAFETY SCREEN RESULTS FOR TANK 241-C-204,  
AUGER SAMPLES 95-AUG-022 AND 95-AUG-023

Date Printed:

AUGUST 24, 1995

2-1

WHC-SD-WM-DP-115, REV. 1

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This Document consists of pages 2-1 through 2-35 and pages 2-2, 2-5, 2-26 and 2-31 were intentionally left blank.

WHC-SD-WM-DP-115, REV. 1

WHC-SD-WM-DP-115, REV. 0-A

NARRATIVE



WHC-SD-WM-DP-115, REV. 1

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90-DAY SAFETY SCREEN RESULTS FOR TANK 241-C-204,  
AUGER SAMPLES 95-AUG-022 AND 95-AUG-023

## ANALYTICAL SUMMARY

Two auger samples from tank 241-C-204 (C-204) were submitted to the 222-S Laboratories for safety screening analyses. Primary results from these analyses were reported previously [1]. As the samples submitted for primary analyses exceeded the screening limit for energetics stated in the Tank Characterization Plan (TCP) [2], secondary analyses were prescribed. Selection of secondary analyses was closely coordinated with safety program personnel, as only limited archive material remained from the sampling event.

Two samples were submitted for Total Organic Carbon (TOC) determination. Total Inorganic Carbon (TIC) was also determined as an artifact of the TOC analytical procedure. The TOC tests indicate that the TOC content of the samples was extremely high (around 13% on an uncorrected wet basis). It is possible that the samples were contaminated by the rag that was caught in the auger samples. Since the results were above the TOC action limit stated in the TCP [2], notifications were made to the appropriate Tank Farms Operations and Safety Program personnel.

An additional Differential Scanning Calorimetry (DSC) analysis was performed on rag material collected from 95-AUG-023. This test was performed in an attempt to better interpret the unusual results reported on the primary analyses (DSC scans had not returned to baseline at the temperature limit of the test). The test results were inconclusive, but did indicate that the rag material reacted sluggishly when analyzed under a nitrogen atmosphere. The scans trended upward with a fairly constant slope up to the temperature limit of the test (500 °C).

One sample was submitted for adiabatic calorimetry by the Reactive System Screening Tool (RSST) procedure. The RSST run, performed on a dried sample, demonstrated that the waste will not propagate a reaction. The thermal response of the sample was very sluggish. The RSST results will be interpreted further once heat capacity tests and a DSC analysis of the post-run sample are performed. These discussions will be included in a subsequent report.

## SCOPE

This document reports the results of secondary analyses performed on auger samples from tank C-204 (95-AUG-022 and 95-AUG-023). Both samples were taken from riser 7 of the tank as no other risers were available for sampling. The TOC and TIC tests, the RSST test, and the DSC analysis on the rag sample are reported.

One sample has been submitted for organic speciation at the Pacific Northwest Laboratory's 329 building. Results of this analysis will be included in the final report, along with any additional tests performed to help interpret the RSST results.

## ANALYTICAL RESULTS

### Total Organic Carbon (TOC)

Two samples were submitted for TOC analysis - the lower half-segment subsample from 95-AUG-022 (sample S95T000961) and the whole segment sample from 95-AUG-023 (sample S95T000963). The TOC analyses were performed by hot persulfate oxidation (procedure LA-342-100, Rev. A-0). The analyses were performed in duplicate. The results for these tests averaged approximately 127,000 micrograms carbon per gram ( $\mu\text{g C/g}$ ) on an uncorrected wet basis. The results are presented in Table 1 and in the data summary tables.

The results greatly exceeded the limit of 30,000  $\mu\text{g C/g}$  stated in the TCP [2]. The limit corresponds to a TOC content of 3 weight percent on a dry basis. When the C-204 samples are corrected for moisture, the dry basis results (presented in Table 1) are about ten times the limit.

Recovery of the standard run with these samples was 92.67%, which is within the 90-110% range specified in the TCP [2]. However, the relative percent difference (RPD) between sample and duplicate results exceeded the 10% criterion for both samples. Sample S95T000961 was run initially two other times, with the results outside the desired upper range of the detector, requiring smaller sample sizes (and not reported). Sample S95T000963 was run an extra time due to the high RPD (44.3%). The triplicate result was 103,000  $\mu\text{g C/g}$ . This result is not included in the data summary tables, but is given in Table 1. Additional reruns are not necessary as it is evident that the samples are far over the limit. The spike performed on sample S95T000961 exhibited a recovery of 119%, which was within the internal laboratory quality control limits of 75-125%.

As both auger samples from the tank encountered a rag apparently discarded into the tank, it is possible that the samples were contaminated with rag fibers or material from the rag (e.g. oil or grease). These results are many times higher than any other tank waste samples tested to date (other than organic phase samples), suggesting that contamination is likely. However, the technicians performing the tests did not report observing any fibers in the small subsamples taken for the TIC/TOC analyses. Samples from tank 241-C-201, which has a similar waste transfer history, averaged 41,700 micrograms TOC per gram [3], suggesting that at least part of the TOC evident in the C-204 samples is attributable to the tank waste.

Table 1. Adjusted Dry TOC Results

Subsample Portion, Auger, and Sample Number	Sample Result ( $\mu\text{g C/g}$ )	Duplicate Result ( $\mu\text{g C/g}$ )	Triplicate Result ( $\mu\text{g C/g}$ )	Mean ( $\mu\text{g C/g}$ )	Moisture Content* (% water)	Adjusted Dry Mean Result ( $\mu\text{g C/g}$ )
Lower Half 95-AUG-022 S95T00961	148,500	130,000	-	139,300	55.70	314,300
Whole Segment 95-AUG-023 S95T00963	91,800	144,000	103,000	112,900	58.00	268,900

\*Moisture content taken from the corresponding thermogravimetric analysis found in Reference 1.

### Total Inorganic Carbon (TIC)

TIC analyses were performed on and reported for the same samples submitted for TOC analysis. The procedure used (LA-342-100) requires removal of the inorganic carbon in order for TOC to be determined. A triplicate analysis was performed on sample S95T00963 (to improve reproducibility of TOC results). The triplicate result of 12,400  $\mu\text{g}$  TIC/g is not included in the data summary tables. All other results are included in the data summary tables. The suspected contamination with rag material is not likely to affect the TIC results, as the rag fibers would consist of organic carbon rather than inorganic carbon. Nonetheless, the results should only be used with caution.

The TIC spike recovery was 77.30 for sample S95T00961, which is within the laboratory's acceptable internal quality control range of 75-125%. The standard run with the samples exhibited a recovery of 90.00%.

### Differential Scanning Calorimetry

A subsample of rag material from auger sample 95-AUG-023 was submitted for DSC analysis in an attempt to help interpret the unusual DSC results from primary analyses [1]. The rag was tested in duplicate under a nitrogen atmosphere using procedure LA-514-113, Rev. B-1. The raw data scans (attached) show an endotherm from ambient temperature to between 250-300  $^{\circ}\text{C}$ , after which the scans trend upward with a fairly constant slope to 500  $^{\circ}\text{C}$  (the temperature limit of the test). The official sample and duplicate test results, presented in the data summary tables, are that no exotherms were detected. The scans are trending upward, but no exotherm can be calculated as the slope is fairly constant and the scans do not return to baseline. The reaction of the rag material under nitrogen is evidently sluggish. This behavior is similar to the results from the primary analyses on the tank waste samples given in Reference 1, in that the scans have not returned to baseline at the temperature limit of the test. It is still unclear whether this behavior is attributable to rag material contaminating the earlier samples, or possibly a synergistic effect of the rag (organic carbon fuel) and the waste (nitrate/nitrite oxidant).

### Adiabatic Calorimetry (by RSST)

The RSST test is described in test plan WHC-SD-WM-TP-104 [4], with calculations performed as outlined in WHC-SD-WM-DTR-026 [5]. The RSST analysis quantifies the exothermic or endothermic behavior of the sample as a function of time and temperature. The test attempts to maintain adiabatic conditions by adding heat to a bomb calorimeter to compensate for sample heat losses. Approximately 10  $\text{cm}^3$  of dried, pulverized sample is placed in the calorimeter. A heater heats the sample at a slow, constant rate until self heating from the exothermic reactions begin to dominate and the reaction goes to completion. By measuring the rate of temperature change, the total temperature change, and the heat capacity of the sample, the chemical rate kinetics and total exothermic energy may be calculated. The rate and amount of non-condensable gases evolved may also be determined.

One sample from auger 95-AUG-022 was analyzed by RSST. A detailed discussion of the results is presented in Appendix A. Further interpretation of the RSST results will be included in a subsequent report (additional DSC runs will be performed to determine sample heat capacity and verify that the exothermic potential of the sample was exhausted).

#### REFERENCES

- [1] J. M. Conner, 1995, *45-Day Safety Screen Results for Tank 241-C-204, Auger Samples 95-AUG-022 and 95-AUG-023*, WHC-SD-WM-DP-115, Rev. 0.
- [2] R. D. Schreiber, 1995, *Tank 241-C-204 Tank Characterization Plan*, WHC-SD-WM-TP-307, Rev. 0.
- [3] R. D. Schreiber, 1995, *45-Day Safety Screen Results for Tank 241-C-201, Auger Samples 95-AUG-025 and 95-AUG-026*, WHC-SD-WM-DP-116, Rev. 0.
- [4] D. B. Bechtold, 1992, *Laboratory Test Plan for Adiabatic Calorimetry of Single and Double Shell Tank Wastes*, WHC-SD-WM-TP-104, Rev. 0.
- [5] D. L. Herting, 1992, *Laboratory Characterization of Samples Taken in December 1991 - Window E - from Hanford Waste Tank 241-SY-101*, WHC-SD-WM-DTR-026, Rev. 0.

## APPENDIX A

### RESULTS OF ADIABATIC CALORIMETRY ON SAMPLE S95T000962, JAR 7268 OF TANK C-204 AUGER MATERIAL

D. B. Bechtold

#### SUMMARY

Jar 7268 contents were dried for several weeks in the draft of 11A hot cell to reduce the moisture content from 57% down to 26% (by TGA), whereupon the contents consisted of brown granular solids and partially decomposed rags. 8.84 grams of granular material was subjected to RSST analysis to 450°C under 7 barg nitrogen, yielding a complex, sluggish self-heating response which never exceeded 6 °C/min (uncorrected), and a total noncondensable gas production of 0.0018 mole/gram. Indications were positive but not complete to verify that the sample had exhausted its self-heating ability at 450 °C.

After the test, the sample had been reduced to a black, unfused granular material reminiscent of charcoal, and a moderate amount of condensate. The Jar 7268 dried material and the post-test material were submitted for DSC-TGA to aid in data interpretation. However the laboratory has discovered an air leakage problem in the DSC and TGA nitrogen supply, and will provide results at a later date.

Correction of the thermal data for  $\phi$  factor, verification of sample exhaustion and calculation of total exothermic energy content must await determination of sample heat capacity from future DSC results. In the meantime, a means to estimate the energy release is provided based on arbitrary heat capacity estimates.

#### PERFORMANCE OF WORK

Work was performed according to WHC-SD-WM-TP-104, Rev. 0 and calculations were made as outlined in WHC-SD-WM-DTR-026, Rev. 0. Sample treatment and experimental data were logged in WHC-N-1014-1, pg. 27 to 31, and separate hardcopies of instrumental data were retained to back up electronic copies.

#### SAMPLE TREATMENT

The sample to be run was dried in jar 7268 in the 11A hot cell for a few weeks by leaving the lid off, and occasionally heating on a hot plate to 60°C. The resulting dried material consisted of a brown, granular solid and dark, balled up, partially decomposed rag pieces. The granular material was selectively loaded into the RSST bulb and the rag pieces were excluded. The RSST aliquot net weight was 8.84 grams, leaving granular material left over for more DSC work, including heat capacity measurements. The RSST bulb was then placed in

the containment bomb, and the bomb was flushed twice with 7 barg. of nitrogen, before filling to nominal 7 barg. nitrogen for the test. The sample was heated in the RSST at a selected rate of 1 °C/min.

After the test, the sample was observed to have converted into an unfused, black granular material reminiscent of charcoal, and a moderate amount of condensate was present on the sheath and containment surfaces. This condensate felt lubricative to the touch, suggesting it may not have been all water.

The pre-test, jar 7268 material was submitted for further DSC and TGA work (lab # J2632), as was also the post-test, charcoal-like material (J2631). This is to include the measurement of heat capacity by DSC and verify the completion of exothermic reaction in the RSST in order to calculate a total sample exothermic energy content. The verification of exothermic energy reaction may be complicated by the tendency of charcoal-like materials to absorb oxygen irreversibly, releasing CO<sub>2</sub> exothermically upon heating. The TGA's produced thus far (J2632) average 25.88% moisture to 200°C.

## THERMAL RESULTS

Figures A-1 to A-3 comprise the thermal data from the test, and the results are tabulated in Table A-2. Figures A-2 and A-3 indicate the detailed thermal response. At 50°C the heat rate departed from the selected baseline slightly, most likely due to non-exothermic changes in sample moisture content and subsequent heat capacity. A minor endotherm was traversed at approximately 160°C followed by an apparent self-heating event which was completed -- or interrupted by endotherms -- at 260-280°C. Another self-heating event began at 280°C, was interrupted at 300°C by a sharp endotherm (which caused a slight increase in heater offset), but continued with additional interruptions or transitions past 400°C, beginning to level off in rate due to exhaustion of reactants.

The heater maximized at 412°C, leaving no more power available to compensate for heat losses and/or provide a steady heat rate baseline above that temperature. The sample continued to self-heat onward towards 435°C at a decaying rate due to some combination of increasing lack of compensation and reactant exhaustion. At 435°C, the rate plummeted due to virtually complete reactant exhaustion before the heater was turned off at 449°C.

This interpretation of the latter stages of the test is subject to some uncertainty. Therefore, due to the proximity of the reactant exhaustion with the maximum temperature of the RSST (45°C), it will be important to verify the lack of further exothermic energy content in the post-test sample by DSC before accepting its calculated value to 450°C from the RSST test.

The fitted Arrhenius parameters for the two self-heating events compiled in Table A-2 are based on a graphically determined effective baseline of 1.31 °C/min. This value was not determined by fitting, but rather by selection to coincide with the most recent discernible baseline, unaffected by endotherms or exotherms, between 90 and 110°C. The fitted activation energies and temperature-referenced rates are sensitive to this choice.



The  $\phi$  factor appearing in the tables, and defined by the footnote, removes the effects of instrument heat capacity from the data. It is very likely in this case to be less than 1.1 in value. Its value may be determined and final corrections made once the heat capacity of the reacting sample material has been measured.

The exothermic energy content of the sample to 450°C may be calculated from the true  $\Delta T$  and the heat capacity of the sample, once it is known. Until that time, the energy may be estimated for scoping purposes by interpolating Figure A-7 with estimated values of heat capacity. However, it is important to obtain verification by RSST reruns or DSC that no energy is left in the sample before accepting the value to be calculated from Table A-2, due to the proximity of reactant exhaustion to the upper limit of the RSST. This verification can be interfered with by the tendency for charcoal-like materials to irreversibly absorb oxygen and exothermically release  $\text{CO}_2$  on reheating, but the interference will be conservative from a safety assessment standpoint.

## GAS PRODUCTION RESULTS

Figure A-6 was calculated from the instrumental data as suggested in the footnotes to Table A-3 (see WHC-SD-WM-DTR-026), making use of the slope determined in Figure A-5. The heatup rates of Figure A-2 were slow enough to permit the steady-state assumption necessary for the calculations. Figure A-6 was then used to graphically determine the change in moles of noncondensable gas in containment, and thereby the calculated specific gas production.

Table A-3 compares quantities so determined with those computed from the overall pressure data in Table A-1. The comparison is reasonably good, and is consistent with past experience that the auxiliary data usually gives production numbers equal to or less than the real-time-data calculation, because auxiliary data is more vulnerable to slow leakage from the RSST containment bomb. The computed average molecular weights of gases, as usual, suffer from the evolution of condensate which contributes to weight loss but not to pressure.

## CONCLUSIONS

The C-204 auger sample is probably not representative of the tank due to the presence of partially decomposed rag pieces. It begs the question whether these rags may have been soaked with something (oil?) when they were thrown down the riser.

The pre-dried sample excluding rag pieces evolved some condensate which may not have been entirely water. The solids formed a black, unfused, charcoal-like granular material, suggesting the absence of excess nitrate and presence of carbon.

The sample's thermal response was very sluggish and its gas production very low. Indications that its exothermic capacity was exhausted at 450°C are

positive but not completely certain -- DSC in a rigorously inert cover gas will help determine if any residual energy remains.

Calculation of the energy evolved during the RSST run will await heat capacity results from the laboratory.

Table A-1. C-204 Auxiliary Test Data.

Test ID	At start of run sample at ambient T			At end of run sample cooled to ambient T				Comment
	P <sub>0</sub> (barg)	T <sub>0</sub> (°C)	w <sub>0</sub> (g)	P <sub>f</sub> (barg)	T <sub>f</sub> (°C)	w <sub>f</sub> (g)	% w (%Δ)	
950724	6.88	24.8	8.84	7.80	23.2	7.44	-15.8	condensate on sheath decreases % w magnitude

Table A-2. C-204 Self-Heat Results.

Test ID	T <sub>onset</sub> (°C)	T <sub>true</sub> <sup>a</sup> Δ (°C)	Max. dT/dt (uncorr.) (°C/min), at T (°C) <sup>a</sup>	Initial E <sub>a</sub> (KJ/mole)	Initial K (C°/min) at T (°C)	Comments
950724	160		φ x 4.3 °C/min at 234 °C	70.8±3.2	1.16±0.01 at 200°C	1st self-heat event
950724	270		φ x 5.7 °C/min at 404 °C	75.4±2.1	3.76±0.03 at 380°C	2nd self-heat event
950724	160	φ x 117 at 450 °C				Overall ΔT for both events

<sup>a</sup>φ = C<sub>ps</sub>[1+(0.8368/(C<sub>ps</sub>w<sub>s</sub>))]. C<sub>p</sub> in Joule/g/°C, w in grams, s denoting sample.

Table A-3. C-204 Gas Production Results.

Test ID	F <sup>a</sup>	N/w <sub>0</sub> by Δalc. <sup>b</sup> (mol/g)	M <sub>avg</sub> by calc. (g/mol)	Max. (dN/dt)/w <sub>0</sub> (uncorr.) (mol/min/g) at T (°C) or minutes from onset	N/w <sub>0</sub> by auxiliary data <sup>b</sup> (mol/g)	M <sub>avg</sub> by auxiliary data <sup>c</sup> (g/mol)
950724	0.1794	0.00179	88.5	3.2x10 <sup>-5</sup> at 250°C, or 40 min from 1st onset	0.00148	107

<sup>a</sup> PV = NRT<sub>effective</sub>. T<sub>effective</sub> = F x T<sub>sample</sub> + (1 - F) x T<sub>ambient</sub>. F = (dP/dT)<sub>0</sub> x T<sub>0</sub>/P<sub>0</sub> in absolute units.

<sup>b</sup>Noncondensable gases only

<sup>c</sup>High because of condensate solvent that contributes weight loss but not pressure.

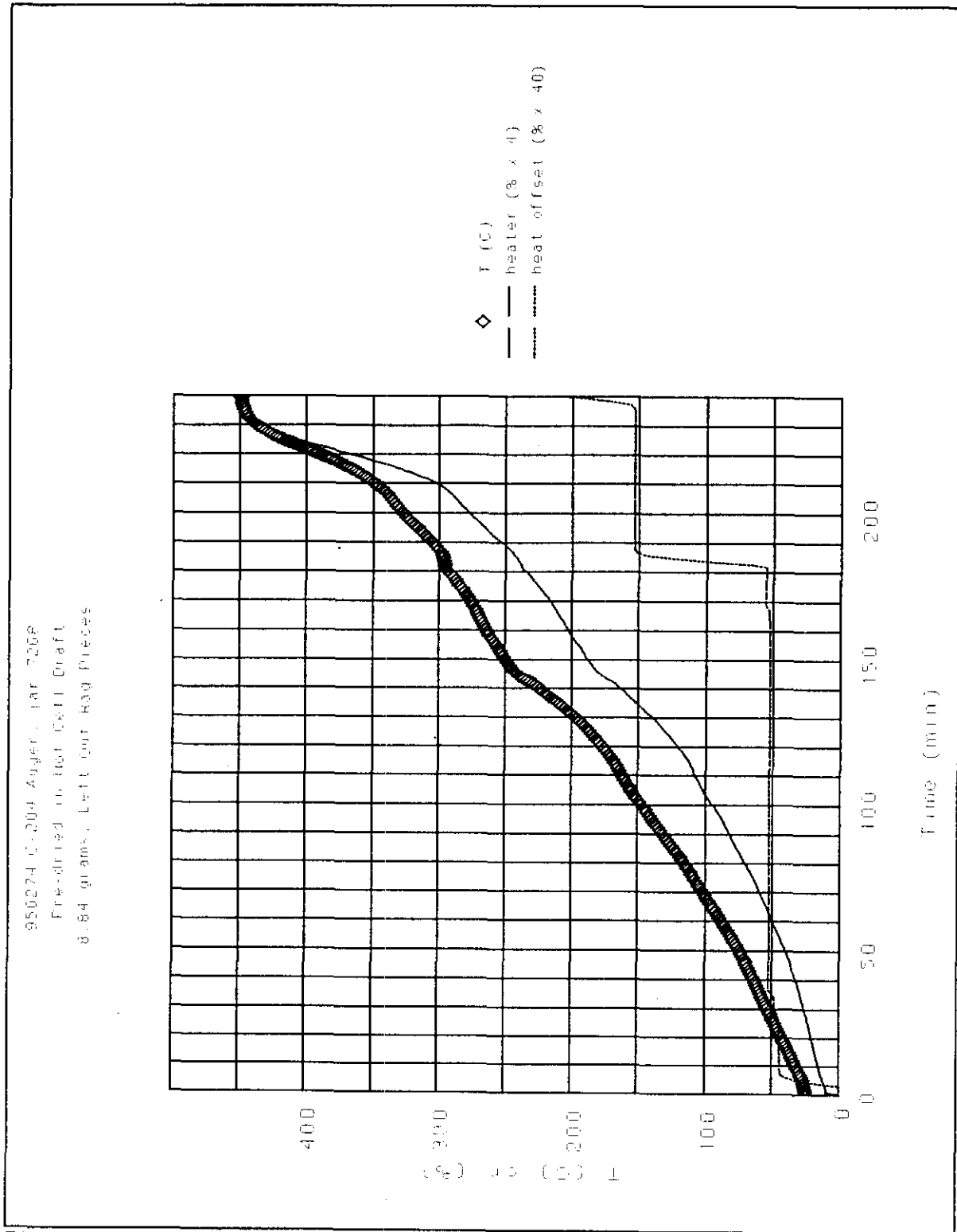


Figure A-1. Temperature vs Time

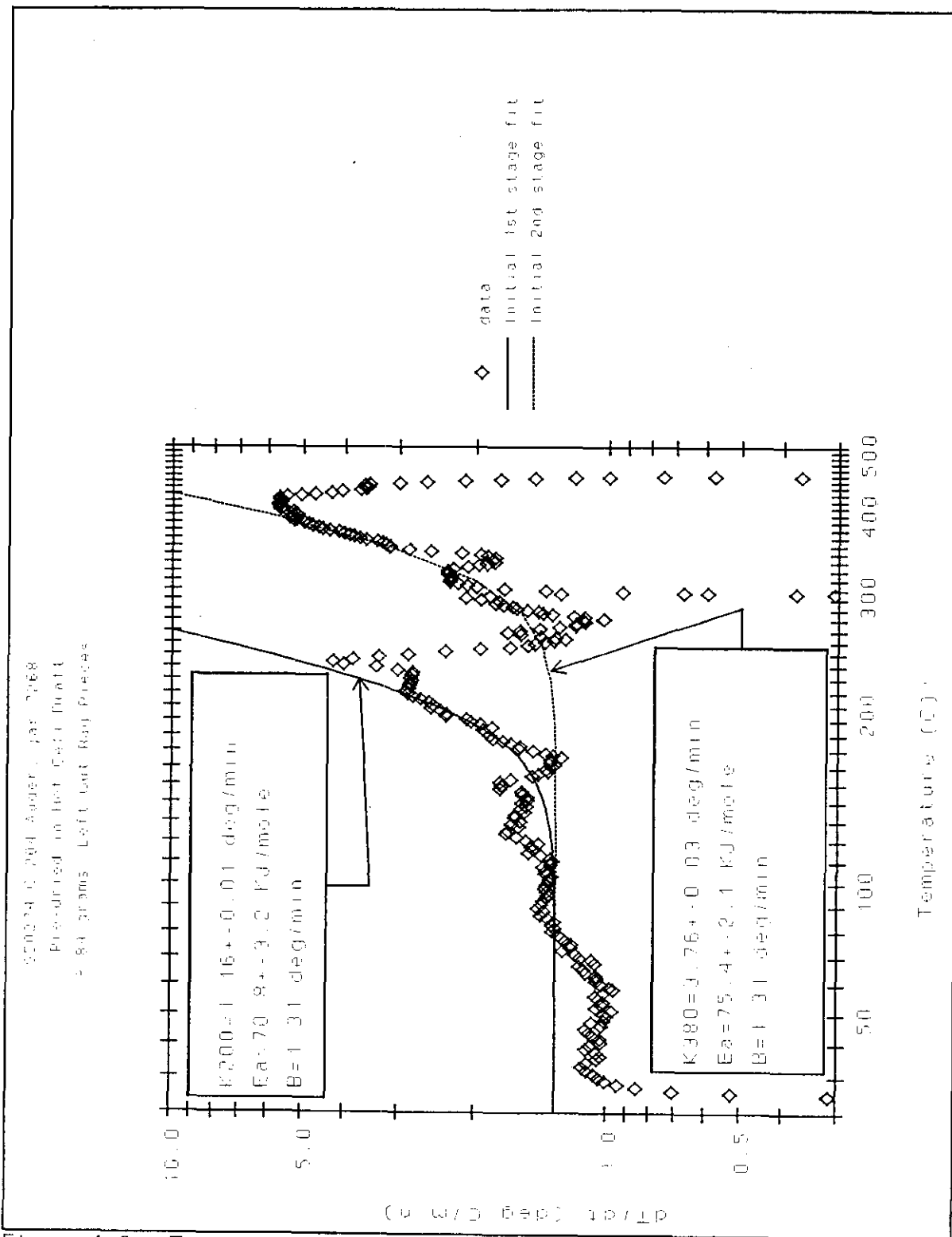


Figure A-2. Temperature Rate vs -1000/Kelvin.

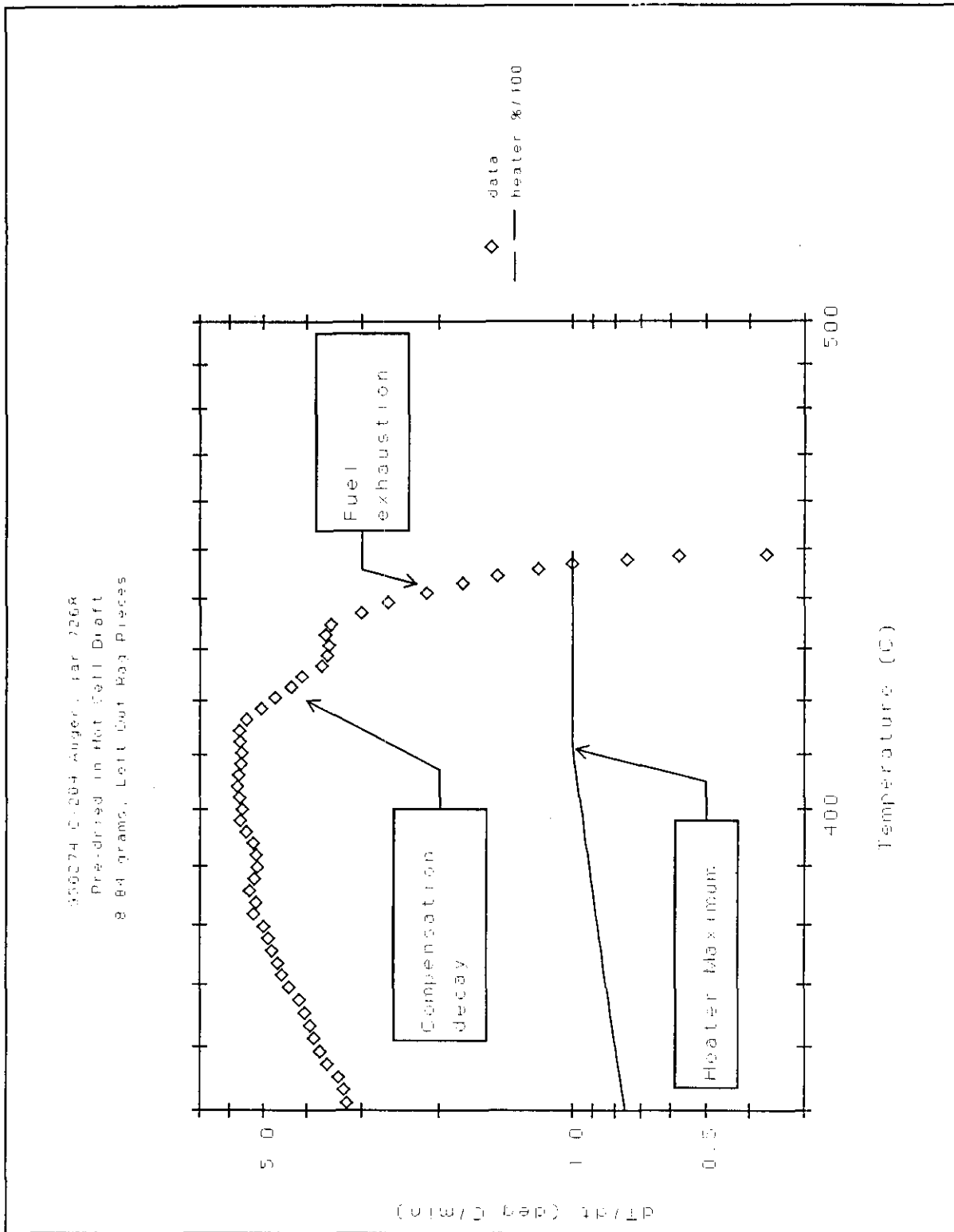


Figure A-3. Detail of Temperature Rate and Heater Power vs  $-1000/\text{Kelvin}$ .

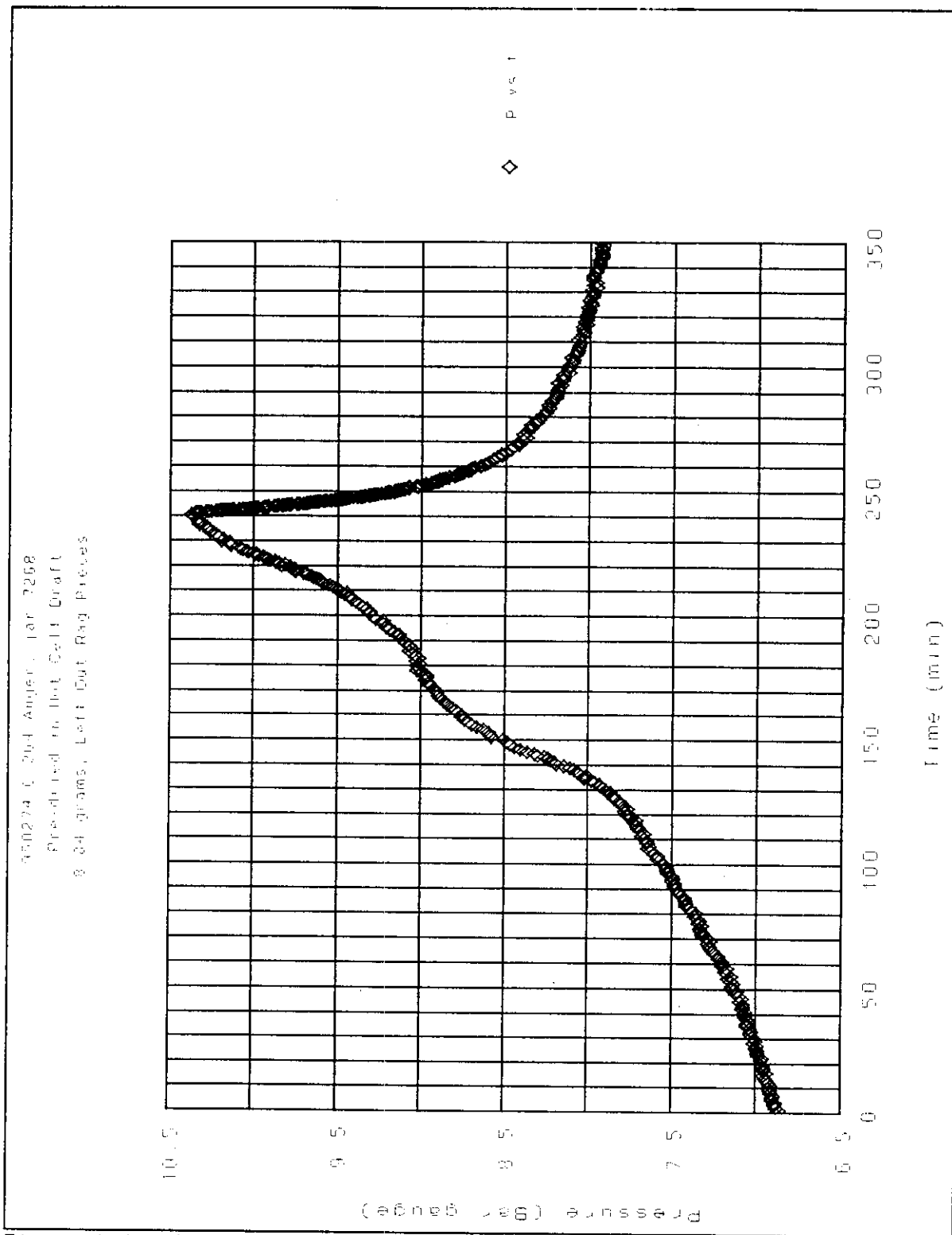


Figure A-4. Containment Pressure vs Time.

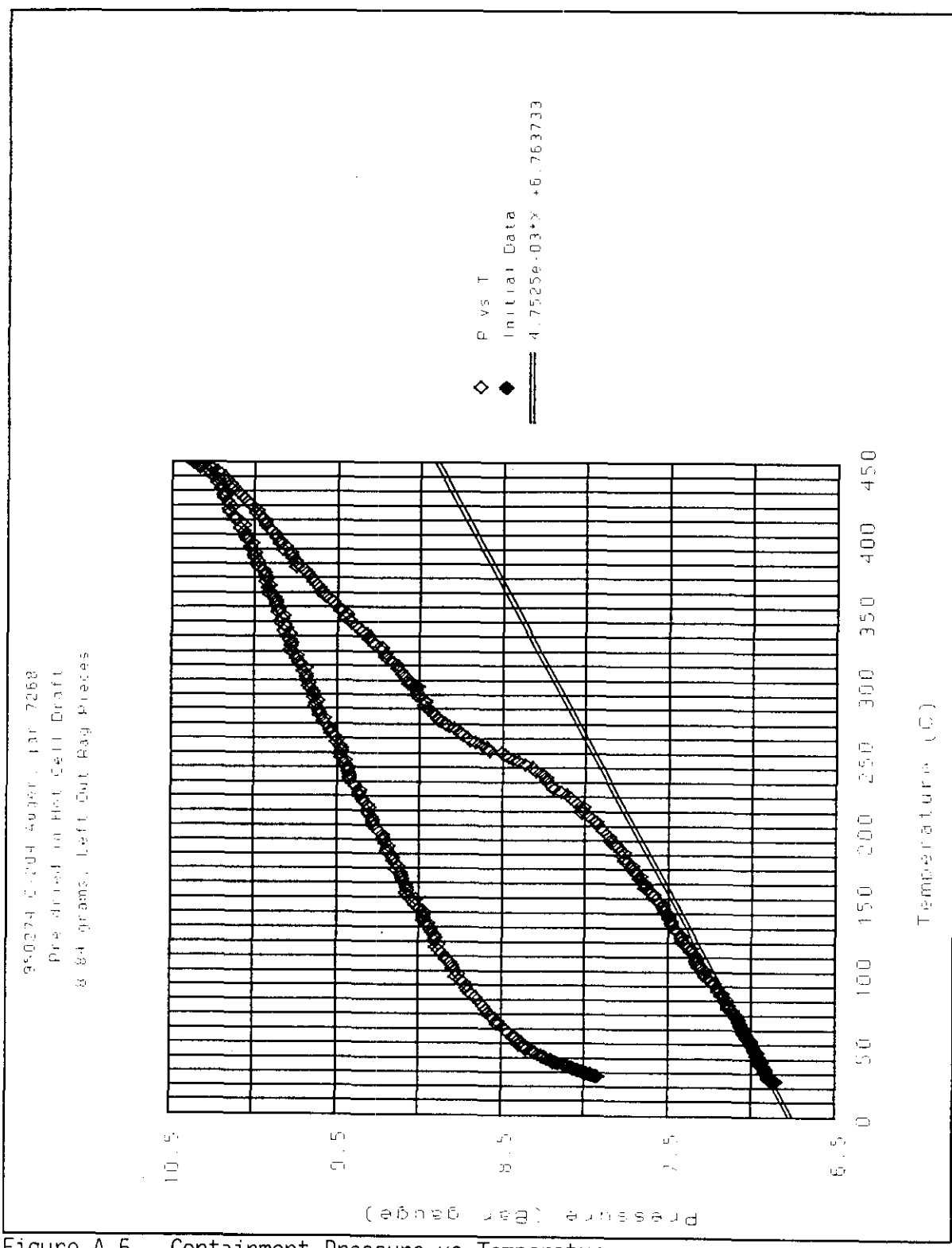


Figure A-5. Containment Pressure vs Temperature.

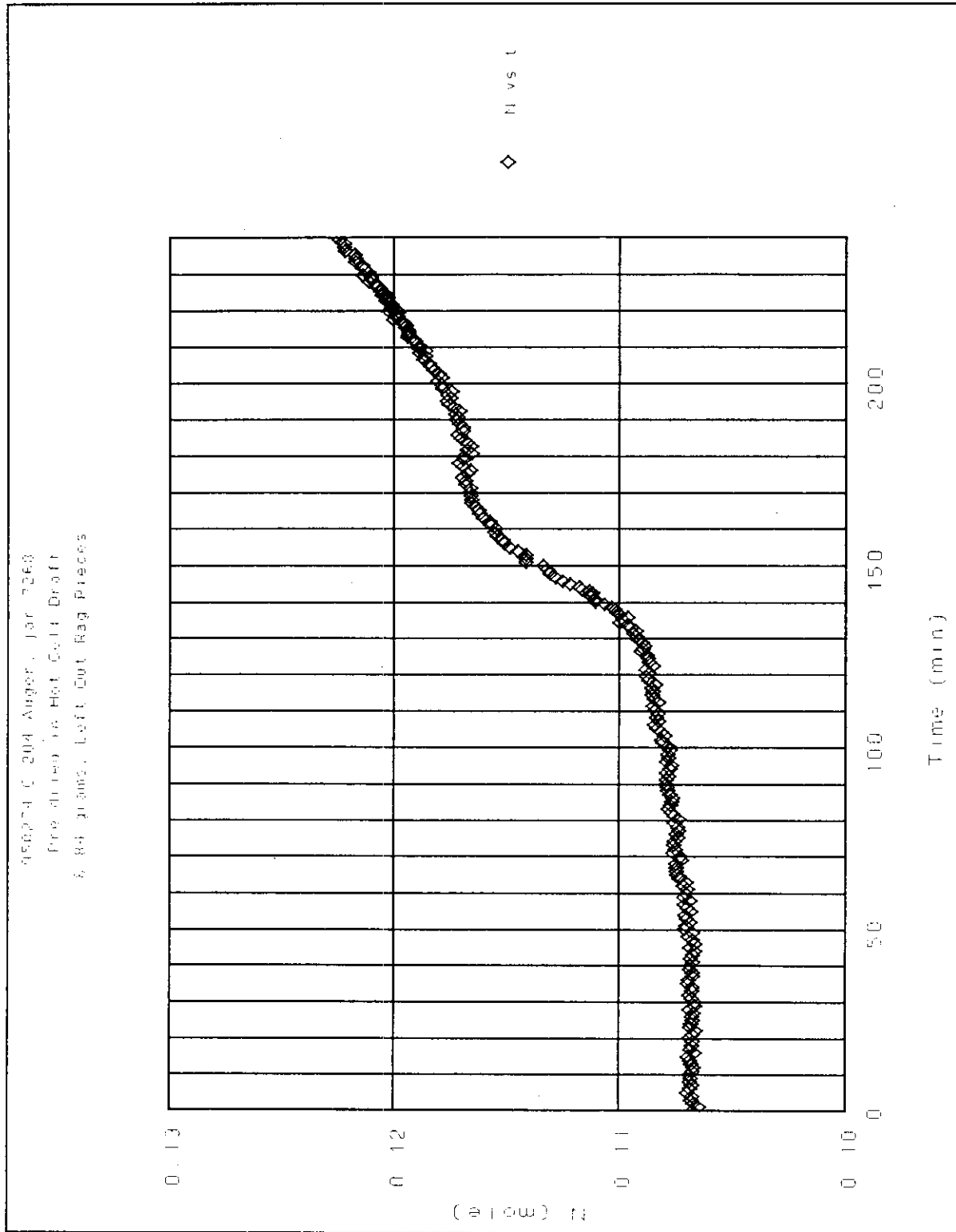


Figure A-6. Moles Noncondensable Gas vs Time.



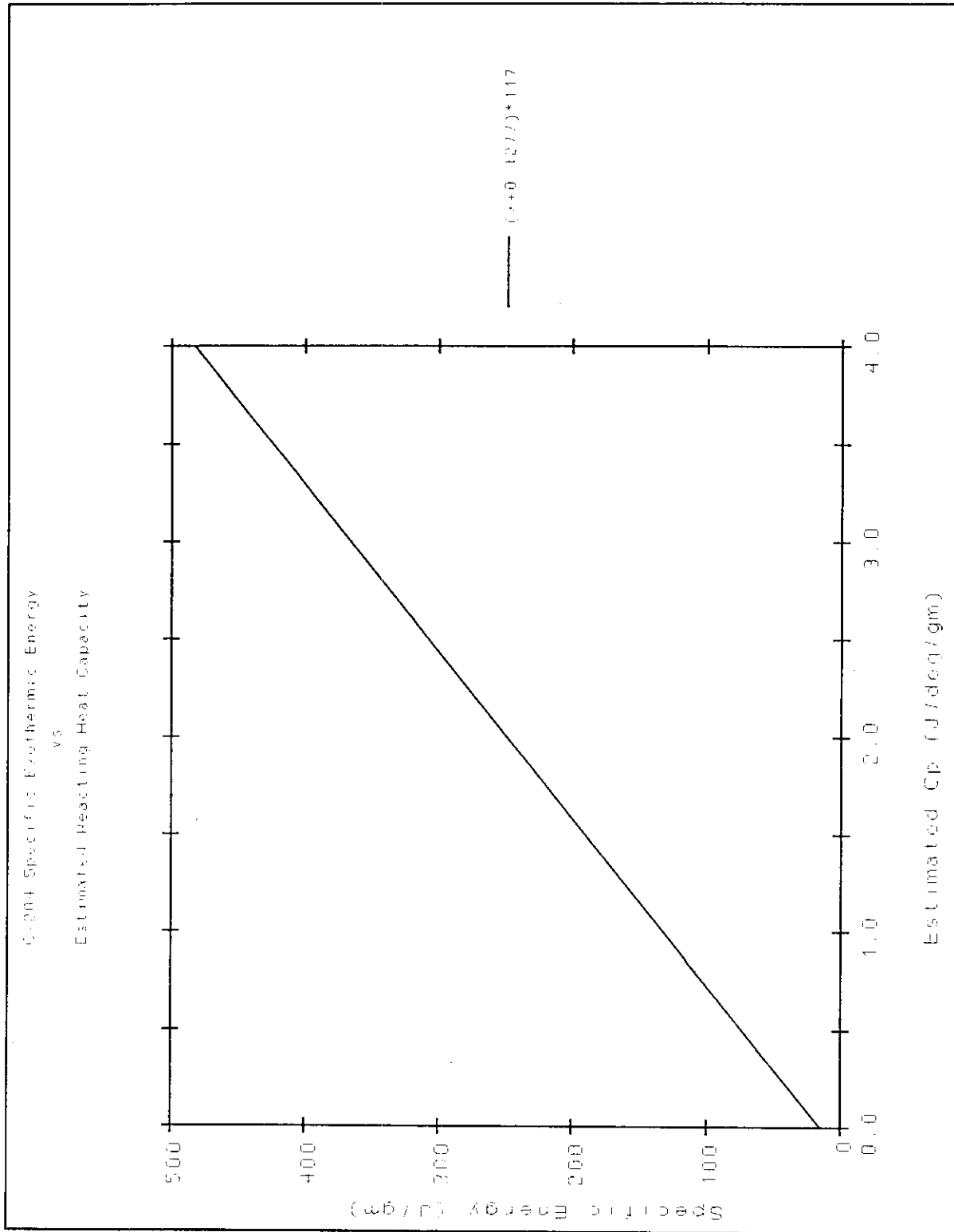


Figure A-7. Total Energy Release vs Estimated Sample Heat Capacity.

CALCULATIONS

C-204 AVERAGE MOISTURE BY TGA:

Original:

$$\begin{array}{r} 58.32\% \\ 50.44 \\ 59.48 \\ 59.19 \\ 58.77 \\ 55.02 \\ 56.39 \\ \hline 397.61 \div 7 = \text{Mean } 56.80\%, \text{ s.d. } = 3.2\% \end{array} \quad (1)$$

Dried, pre-RSST test:

$$\frac{(25.74\% + 26.01\%)}{2} = \text{Mean } 25.875\%, \text{ s.d. } = 0.19\% \quad (2)$$

Post-RSST test (reinterpreted):

$$\frac{1.43\% + 1.49\%}{2} = \text{Mean } 1.46\%, \text{ s.d. } = 0.042\%$$

$\Delta T_{\text{true}}$ :

$$T_i \approx 160^\circ, t_i \approx 108.3 \text{ min}, T_e \approx 450^\circ, t_e \approx 240.4 \text{ min}$$

$$B \approx 1.31^\circ/\text{min},$$

$$\begin{aligned} \Delta T_{\text{true}} &= \phi([T_e - T_i] - B[t_e - t_i]) \\ &= \phi([450 - 160] - 1.31[240.4 - 108.3]) \\ &= \phi 117^\circ \end{aligned} \quad (4)$$

$$\phi = \frac{C_{ps}W_s + C_{ph}W_h}{C_{ps}W_s} = 1 + \frac{0.8368}{C_{ps}8.84[1 - 0.2588]}$$

KINETIC PARAMETERS:

For the first runaway,  $dT/dt$  vs  $Z = -1000/(T+273.15)$  data for temperatures between 170°C and 270°C and times less than 240 minutes are least-squares fit by the function

$$\frac{dT}{dt} = B + K_{200} e^{\frac{E_a}{R}(Z - Z_{200})}, \quad (5)$$

$$\text{where } Z = -\frac{1000}{T + 273.15}, \quad Z_{200} = -\frac{1000}{200 + 273.15}$$

with parameters  $E_a$  (KJ/mole),  $K_{200}$  (°C/min); and constants  $B = 1.31$  °C/min and  $R = 8.3144$  J/mole/°K. For the second runaway, data from 350°C to 385°C at times less than 240 minutes is fit to the analogous function where 380°C is substituted for 200°C.

SPECIFIC EXOTHERMIC ENERGY RELEASE:

$$\begin{aligned} \hat{Q} &= C_{ps} \Delta T_{true} = C_{ps} \phi 117 = C_{ps} \left( 1 + \frac{0.8368}{C_{ps}(1 - 0.2588) 8.84} \right) 117 \\ &= (C_{ps} + 0.1277) 117 \quad \left( \frac{\text{Joule}}{\text{gram}} \right) \end{aligned} \quad (6)$$

TOTAL SPECIFIC NONCONDENSIBLE GAS PRODUCTION:

By Calculated Graph:

$$N_i = 0.1068 \text{ mole}, \quad N_e = 0.1227 \text{ mole}, \quad w_0 = 8.84 \text{ grams}$$

$$\frac{\Delta N}{w_0} = \frac{0.1227 - 0.1068}{8.84} = 0.00179 \frac{\text{mole}}{\text{gram}} \quad (7)$$

By Auxiliary Data:

$$P_0 = 6.878 \text{ barg}, T_0 = 24.78 \text{ }^\circ\text{C}, P_f = 7.80 \text{ barg}, T_f = 23.2 \text{ }^\circ\text{C}$$

$$V = 335 \text{ cm}^3, R = 83.14 \frac{\text{bara cm}^3}{\text{mole } ^\circ\text{K}}$$

$$\begin{aligned} \frac{\Delta N}{w_0} &= \frac{1}{w_0} \frac{V}{R} \left[ \frac{P_f + 1.01325}{T_f + 273.15} - \frac{P_0 + 1.01325}{T_0 + 273.15} \right] \\ &= \frac{1}{8.84} \frac{335}{83.14} \left[ \frac{7.80 + 1.01325}{23.2 + 273.15} - \frac{6.878 + 1.01325}{24.78 + 273.15} \right] \\ &= 0.00148 \frac{\text{mole}}{\text{gram}} \end{aligned} \quad (8)$$

AVERAGE MOLECULAR WEIGHT:

$$\begin{aligned} M_{\text{avg.}} &= \frac{-\Delta w/w_0}{\Delta N/w_0} \\ &= \frac{-(-0.158)}{0.00179} = 88 \frac{\text{gram}}{\text{mole}} \quad \text{by calculated graph} \\ &= \frac{-(-0.158)}{0.00148} = 107 \frac{\text{gram}}{\text{mole}} \quad \text{by auxiliary data} \end{aligned} \quad (9)$$

MAXIMUM UNCORRECTED RATE OF NONCONDENSIBLE GAS PRODUCTION:

From Calculated Graph:

$$\frac{1}{w_0} \left( \frac{dN}{dt} \right)_{\text{max}} \approx \frac{1}{8.84} \left( \frac{0.13 - 0.10}{209 - 103} \right) = 3.2 \times 10^{-5} \frac{\text{mole}}{\text{min gram}} \quad (10)$$

WHC-SD-WM-DP-115, REV. 1

**SAMPLE DATA SUMMARY**

**WHC-SD-WM-DP-115, REV. 1**

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Data Summary Tables: 90-Day Safety Screening Results  
C-204

CORE NUMBER: n/a  
SEGMENT #: 95-AUG-022

SEGMENT PORTION: L Lower Half of Segment

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000961			TOC by Persulfate/Coulometry	ug/g	-1.0e+03	3.0e+04	92.67	35.70	1.48e+05	1.30e+05	1.39e+05	12.9	119.0	80.00	n/a
S95T000961			TIC by Acid/Coulometry	ug/g	-1.0e+03	3.0e+04	90.00	2.600	9.36e+03	8.13e+03	8.74e+03	14.1	77.30	5.000	n/a

=> Limit violated

=> Selected Limit

2-27



WHC-SD-WM-OP-115, REV. 1

Data Summary Tables: 90-Day Safety Screening Results  
C-204

CORE NUMBER: n/a  
SEGMENT #: 95-AUG-023

SEGMENT PORTION: W Whole Segment

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000963			TOC by Persulfate/Coulometry	ug/g	-1.0e+03	3.0e+04	92.67	35.70	9.18e+04	1.44e+05	1.18e+05	44.3	n/a	80.00	n/a
S95T000963			TIC by Acid/Coulometry	ug/g	-1.0e+03	3.0e+04	90.00	2.600	1.38e+04	1.07e+04	1.22e+04	25.3	n/a	5.000	n/a

 => Limit violated  
 => Selected Limit

2-28

WHC-SD-WM-DP-115, REV. 1



Data Summary Tables: 90-Day Safety Screening Results  
C-204

CORE NUMBER: n/a  
SEGMENT #: 95-AUG-023

SEGMENT PORTION: Facie

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000966			DSC Exotherm Dry Calculated	Joules/g Dry	-1.0e+03	481.0	n/a	n/a	0.00e+00	0.00e+00	0.00e+00	n/a	n/a	n/a	n/a
S95T000966			DSC Exotherm using Mettler	Joules/g	-1.0e+03	481.0	102.8	n/a	0.00e+00	0.00e+00	0.00e+00	n/a	n/a	n/a	n/a

=> Limit violated  
=> Selected Limit

2-29

WHC-SD-WM-DP-115, REV. 1

**WHC-SD-WM-DP-148, REV. 1**

**SAMPLE ANALYSES RESULTS**

**WHC-SD-WM-DP-115, REV. 1**

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# LABCORE Data Entry Template for Worklist# 1458

Analyst: SMF Instrument: DSC0 1 Book # 12N14-A

Method: LA-514-113 Rev/Mod B-1

Worklist Comment: Please run the C-204 DSCs under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID	<u>28.45</u>	<u>29.3</u>	<u>N/A</u>	Joules/g
95000069	C-204	2 SAMPLE	S95T000966	0	DSC-01	SOLID	<u>N/A</u>	<u>Ø</u>		Joules/g
95000069	C-204	3 DUP	S95T000966	0	DSC-01	SOLID	<u>Ø</u>	<u>Ø</u>	<u>N/A</u>	Joules/g

Final page for worklist # 1458

Blandina Valenzuela for SM Fulton

Analyst Signature

Date

5/30/95

[Signature]

Analyst Signature

5-30-95

Date

Verified by Blandina Valenzuela 5/30/95

Data Entry Comments:

The thermograms did not display any distinct  
exotherms, however there is a definite upward (exothermic) trend  
seen on the thermogram.

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number,  
R = Replicate Number, A = Aliquot Code.

SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT  
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 233 TO 235.

BEST AVAILABLE COPY

DSC STD 12N14-A

6.748 mg

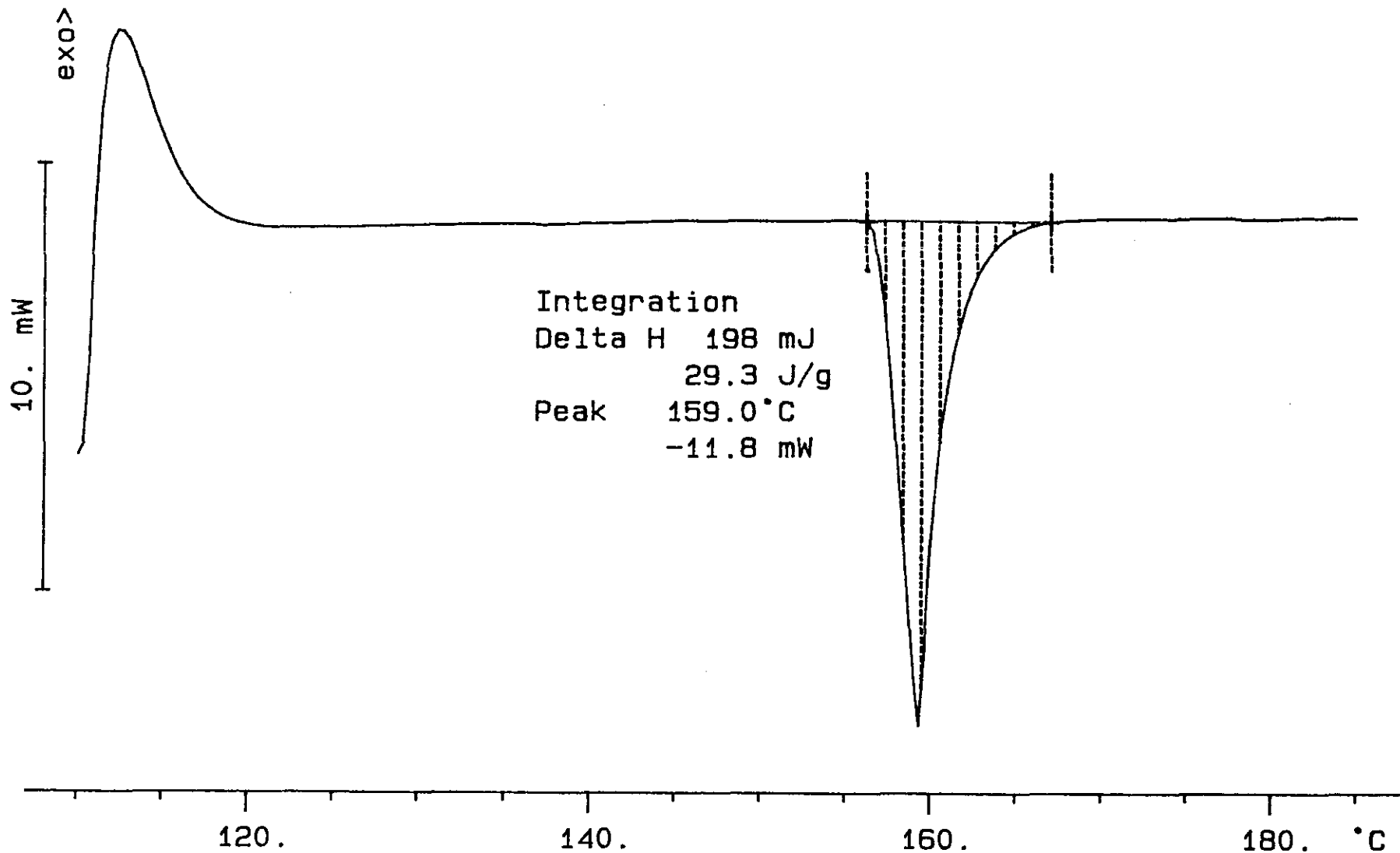
Rate: 10.0 °C/min

File: 00091.001

DSC METTLER 25-May-95

Ident: 0.0

222-S Laboratory



WMC-SD-WM-DP-116, REV. 1

BEST AVAILABLE COPY

S95T000966 N2  
18.954 mg

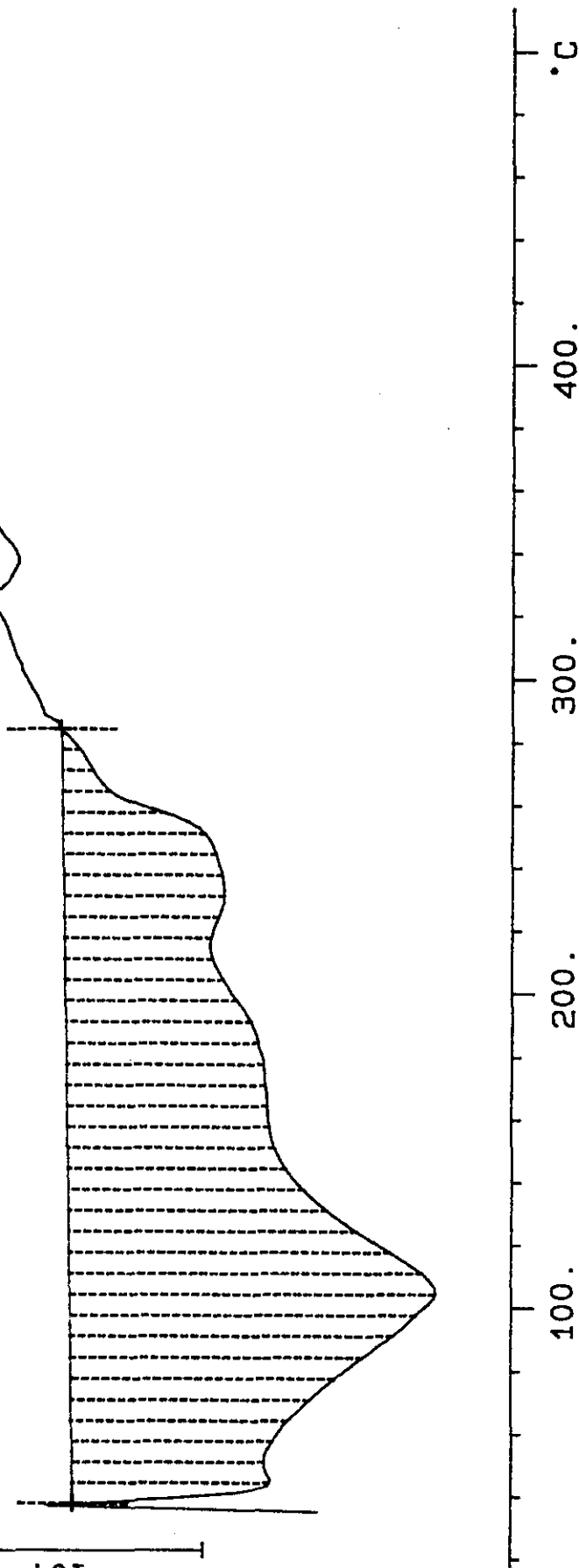
File: 00092.001 DSC METTLER 25-May-95  
Ident: 0.0 222-S Laboratory

Rate: 10.0 °C/min

exo >

Integration  
Delta H 9358 mJ  
493.7 J/g  
Peak 104.7 °C  
-11.6 mW

10. mW



BEST AVAILABLE COPY

S95T000966 (DUP) N2

13.561 mg

Rate: 10.0 °C/min

File: 00093.001

DSC METTLER

25-May-95

Ident: 0.0

222-S Laboratory

exo>

Integration

Delta H 7099 mJ

523.5 J/g

Peak 100.2°C

-9.7 mW

20. mW

100.

200.

300.

400.

500.

°C

WHC-SD-WM-DP-115, REV. 1



Westinghouse  
Hanford Company

WHC-SD-WM-DP- 115, REV. 1

P.O. Box 1970 Richland, WA 99352

PART III

WHC-SD-WM-DP-115, REV. 0

### ANALYTICAL SERVICES

45-DAY SAFETY SCREEN RESULTS FOR TANK 241-C-204,  
AUGER SAMPLES 95-AUG-022 AND 95-AUG-023

Date Printed:

June 14, 1995

**3-1**



**WHC-SD-WM-DP-115, REV. 1**

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This Document consists of pages 3-1 through 3-51 and pages 3-2, 3-5, 3-13 and 3-17 were intentionally left blank.

**WHC-SD-WM-DP-115, REV. 1**

**NARRATIVE**

WHC-SD-WM-DP-115, REV. 1

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WHC-SD-WM-DP-115, Rev. 0

45-DAY SAFETY SCREEN RESULTS FOR TANK 241-C-204,  
AUGER SAMPLES 95-AUG-022 AND 95-AUG-023ANALYTICAL SUMMARY

Two auger samples from tank 241-C-204 (C-204) were received at the 222-S Laboratories and underwent safety screening analysis, consisting of differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and total alpha activity. The three samples submitted for energetics determination by DSC exceeded the notification limit. As required by the Tank Characterization Plan, the appropriate notifications were made within 24 hours of official confirmation that the limit was exceeded. Secondary analyses have been initiated. Results from secondary analyses will be included in a revision to this report.

A rag was caught in both auger samples. The rag material was segregated in the hot cell. None of the chemists nor analysts reported seeing any rag fibers contaminating the samples.

SCOPE

This document serves as the 45-day report deliverable for the tank C-204 auger samples collected on May 2, 1995 (samples 95-AUG-22 and 95-AUG-023). Each sample was received, extruded, and analyzed by the 222-S Laboratories in accordance with the Tank Characterization Plan (TCP) referenced below. Included in this report are the primary safety screening results (DSC, TGA, and total alpha) and copies of all DSC and TGA raw data scans as requested in the TCP. Photographs of the auger samples were taken during extrusion and, although not included in this report, are available.

The results of secondary analyses will be included in a revision to this report. The secondary analyses being conducted are described below.

SAMPLE RECEIPT, EXTRUSION, AND SUBSAMPLING95-AUG-022

Sample 95-AUG-022 was collected from riser 7 (east coordinate) of tank C-204 using a 20-inch auger sampler. The sample was taken on May 2, 1995 at 1030 hours. It was received into the 222-S Laboratories on May 3 and extruded on May 4. Upon extrusion, it was evident that a rag had been caught by the auger. Some tank waste material was retrieved as well. A gob of waste (4.3 g) on flute 8 of the auger was subsampled as upper half solids. The rag material intermixed with waste was on flutes 11 through 18. All other flutes were bare. A total of 158.5 grams of solid material was collected, with no drainable liquid. Of that amount, 104.3 grams were segregated as rag material, 53.8 grams were segregated as lower half solids, and 4.3 grams were subsampled as upper half solids. The tank waste solids appeared dark brown.

It is anticipated that the archive material for this auger (sample S95T000892) will be used up in secondary analyses. Subsamples are identified in Table 1.

### 95-AUG-023

Sample 95-AUG-023 was collected from riser 7 (west coordinate) of tank C-204 using a 20-inch auger sampler. The sample was taken on May 2, 1995 at 1135 hours. It was received into the 222-S Laboratories on May 3 and extruded on May 5. As with sample 95-AUG-022, a rag was caught in the auger. Rag and tank waste material were recovered from flutes 13 through 18 of the auger. All other flutes were bare. A total of 135.0 grams of solid material were collected, with no drainable liquid. Of that amount, 93.9 grams of material were segregated as rag material, and 41.1 grams were segregated as tank waste solids. The tank waste appeared to be a mixture of yellow and dark brown solids. Upon subsampling (and incidental mixing), the material appeared brown. The sample was analyzed on a whole segment basis, as no change in strata could be seen and recovery was low. The archive sample (sample S95T000982) from this auger is expected to be used up in secondary analyses. Subsamples are identified in Table 1.

Table 1. C-204 Subsample Identification

Sample ID	Sample Description	Analyses
S95T000876	95-AUG-022 extrusion	extrusion
S95T000877	95-AUG-023 extrusion	extrusion
S95T000878	95-AUG-022 upper half solids, direct analysis	DSC/TGA
S95T000879	95-AUG-022 upper half fusion	fusion/alpha
S95T000880	95-AUG-022 rag material	archive
S95T000881	95-AUG-022 lower half solids, direct analysis	DSC/TGA
S95T000882	95-AUG-022 lower half fusion	fusion/alpha
S95T000883	95-AUG-022 upper half auger subsample	subsampling
S95T000884	95-AUG-022 lower half auger subsample	subsampling
S95T000885	95-AUG-022 lower half archive	secondary analyses
S95T000888	95-AUG-023 rag material	archive
S95T000890	95-AUG-023 whole segment solids, direct analysis	DSC/TGA
S95T000891	95-AUG-023 whole segment fusion	fusion/alpha
S95T000892	95-AUG-023 whole segment archive	secondary analyses
S95T000893	95-AUG-023 whole segment auger subsample	subsampling

## ANALYTICAL RESULTS

Analytical results are summarized in Tables 5 and 6, with the applicable notification limits shaded. For tests where more than one replicate was performed, the results are presented in a another table for clarity (e.g. Tables 2, 3, and 4). The summary tables (created electronically from the laboratory sample management program) only include sample and duplicate results.

### DSC (Energetics Content)

DSC analyses were performed under a nitrogen atmosphere using procedure LA-514-113, Rev. B-1. Exotherms exceeding the notification limit of 481 J/g were detected for all three samples. Safety program personnel were consulted for direction in running secondary analyses. The secondary analyses being conducted are discussed below.

Three LMCS control standards were run along with these samples, exhibiting recoveries ranging from 103.7 to 107.9 percent, all within the program's specified accuracy control limits of 90 to 110 percent recovery.

**Results for S95T000890.** The sample and duplicate results for sample S95T000890 (from 95-AUG-023) were 952.1 and 665.7 J/g respectively (on a dry weight basis). The relative percent difference (RPD) between sample and duplicate results was 35.4%. As this result was not within the TCP target of 10%, a triplicate sample was analyzed, with a result of 822.9 J/g. The triplicate result is not included in the summary tables, but is shown in Table 2 below. The scans for the sample and triplicate results appear similar in shape. The mean of the three results is 813.6 with a standard deviation of 143.4. Several factors could have contributed to this variability - the small sample size used for the DSC (typically 15-35 milligrams), the high moisture content of these samples, or insufficient homogenization. Also, contamination of rag material is a possibility (all visible rag material was segregated in the hot cell; however, individual rag fibers could have remained. None of the chemists nor analysts reported seeing fibers contaminating these samples). The DSC results for sample S95T000890 are presented in Table 2 and Table 6.

Table 2. Summary of DSC Results for S95T000890

Sample	Result (J/g)	Duplicate	Triplicate	Mean	Std. Dev.
S95T000890	952.1	665.7	822.9	813.6	143.4

**Results for S95T000878 and S95T000881.** Reproducible results for samples S95T000878 and S95T000881 were not obtained. The exotherms continued through 500°C (a baseline was not re-established). A modification to the analysis was initiated to increase the temperature limit to 600°C. Even in this case, the exotherms continued without no clear baseline at the limit of the test. The standards which were run with these samples exhibited acceptable recovery

(within 10% of the accepted true value). The empirical observation is that this is a real event. Safety program personnel were consulted when these high exotherms were observed, resulting in a selection of a suite of secondary analyses (discussed below).

The DSC analyzer can only integrate between fixed points on the graph; therefore, since the scans did not return to baseline, these data can only be reported as minimum values. Both samples (S95T000878 and S95T000881) were run in triplicate. The largest exotherm on sample S95T000878 was >1234.0 J/g (dry basis) on the duplicate analysis. The largest exotherm on sample S95T000881 was >1149 J/g (dry basis) on the triplicate analysis. As the scans for these samples did not return to baseline, the RPDs calculated in Table 5 are not applicable. The sample mean and standard deviation were also not calculated for these samples because only "greater than" values were obtained.

The endotherms for these samples were also quite large, dominating the scans up as far as 300°C. In an attempt to isolate the exotherms, subsamples from S95T000878 and S95T000881 were preheated to approximately 240°C by TGA (temperature raised at a rate of 10°C per minute) to remove water from the samples. The subsamples were then analyzed by DSC. These runs, marked as "test," can only be considered unofficial results. The result for S95T000878 was >1977.1 J/g and for S95T000881 was >962.4 J/g. The DSC results for samples S95T000878 and S95T000881 are presented in Table 3.

Table 3. Summary of DSC Results for S95T000878 and S95T000881

Sample	Result	Duplicate	Triplicate	"Test"	Mean	Std. Dev.
S95T000878	>445.6	>1234.0	>696.5	>1977.1*	n/a	n/a
S95T000881	>647.3	>76.1	>1149.0	>962.4*	n/a	n/a

\*"test" results are unofficial and should only be used with caution.

### TGA (Moisture Content)

Weight percent water is calculated from weight loss by TGA. These analyses were performed under a nitrogen atmosphere using procedure LA-560-112, Rev. A-2. Results for the three samples and their duplicates ranged in value from 50.44 to 59.92 percent water by weight. Results for sample S95T000878 exceeded the RPD target of 10%. A triplicate analysis was performed, resulting in a determination of 59.48 weight percent water, compared to 58.32% and 50.44% for the sample and duplicate. The mean of the three results for sample S95T000878 is 56.08, with a standard deviation of 4.92. The TGA results for sample S95T000878 are presented in Table 4.

All TGA results were well above the safety screening minimum of 17 weight percent. Three LMCS control standards were run with these analyses, exhibiting recoveries ranging from 99.76 to 100.7 percent, which were within the program's specified accuracy control limits of 90 to 110 percent.



Table 4. Summary of TGA Results for S95T000878

Sample	Result (wt% H <sub>2</sub> O)	Duplicate	Triplicate	Mean	Std. Dev.
S95T000878	58.32	50.44	59.48	56.08	4.92

Total Alpha Activity

Analyses for total alpha activity were performed on three samples. Samples were prepared by fusion using procedure LA-549-141, Rev. C-3, and analyses were performed using procedure LA-508-101, Rev. D-2. A sample duplicate was performed on each sample. Sample and duplicate results ranged from 0.00643 to 0.0519  $\mu\text{Ci/g}$ . The RPDs for samples S95T000879 and S95T000882 exceeded the TCP target of 10%. Since none of the results were more than ten times the detection limit, the variability is expected. As all results were below the safety screening limit of 41  $\mu\text{Ci/g}$  by a factor of approximately 800 or more, reruns were deemed unnecessary.

Two control standards were run, with recoveries of 105.7 and 90.5%, both within the TCP target of 90 to 110%. A spike was performed on sample S95T000879, with a recovery of 61.9%. This is outside of the TCP target recovery of 90 to 110%. Spike recoveries for alpha have typically been below the target criterion. The laboratory is proposing several minor changes to the methodology for this test to improve recovery in some cases. Since the sample results were far below the action limit, the poor spike recovery did not necessitate further testing (this method is for screening purposes - highly accurate results are not required far below the limit).

Secondary Analyses

Planning for secondary analyses was initiated once the exotherms exceeding the DSC criterion were observed. The strategy for secondary analyses was coordinated closely with safety program personnel. Conservation of sample was critical as only small archive samples remained after primary analyses. Two subsamples were submitted for Total Organic Carbon (TOC) determination per the TCP. These results will quantify the amount of organic material in the tank. The cyanide analyses were waived as the history of the tank did not include transfers of ferrocyanide streams. One remaining archive sample will be prepared for adiabatic calorimetry (by a method termed Reactive System Screening Tool). This method is called for as a secondary analysis in the TCP. The RSST result will provide a better understanding of the potential for propagating chemical reactions than the DSC. The final archive sample will be prepared for shipment to PNL for organic speciation. This will identify the organics present in the sample causing the high exotherm, as well as provide useful data for waste aging studies.

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This will consume all remaining tank waste samples from tank C-204 (the segregated rag material will be retained for a period of time in the hot cell). Results of secondary analyses will be included in a revision to this report.

Responsible Project Coordinator: J. M. Conner

REFERENCE Schreiber, R. D., 1995, WHC-SD-WM-TP-307, Revision 0, "*Tank 241-C-204 Tank Characterization Plan*", dated March 6, 1995.

**WHC-SD-WM-DP-115, REV. 1**

**SAMPLE DATA SUMMARY**

**WHC-SD-WM-DP-115, REV. 1**

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Summary Tables - Preliminary Safety Screening Results  
C-204

CORE NUMBER: n/a  
SEGMENT #: 95-AUG-022

TABLE 5

SEGMENT PORTION: U Upper Half of Segment

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000878			% Water by TGA using Mettler	%	17.000	110.000	100.3	n/a	58.32	50.44	54.38	14.5	n/a	n/a	n/a
S95T000878			DSC Exotherm Dry Calculated	Joules/g Dry	-999.000	481.010	n/a	n/a	> 445.6	>1234.0	n/a	n/a	n/a	1.00e-04	n/a
S95T000878			DSC Exotherm using Mettler	Joules/g	-999.000	481.010	107.9	n/a	>195.7	>542.0	n/a	n/a	n/a	n/a	n/a
S95T000879	F		Alpha of Digested Solid	uCi/g	-999.000	41.010	90.54	<2.81e-03	6.43e-03	1.45e-2	1.05e-02	77.1	61.90	7.00e-03	73.8

L Lower Half of Segment: L Lower Half of Segment

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000881			% Water by TGA using Mettler	%	17.000	110.000	100.3	n/a	55.02	56.39	55.70	2.46	n/a	n/a	n/a
S95T000881			DSC Exotherm Dry Calculated	Joules/g Dry	-999.000	481.010	n/a	n/a	> 647.3	>76.1	n/a	n/a	n/a	1.00e-04	n/a
S95T000881			DSC Exotherm using Mettler	Joules/g	-999.000	481.010	107.9	n/a	>286.7	>33.4	n/a	n/a	n/a	n/a	n/a
S95T000882	F		Alpha of Digested Solid	uCi/g	-999.000	41.010	90.54	<2.81e-03	2.34e-02	1.21e-2	1.78e-02	63.7	n/a	7.00e-03	33.3

=> Limit violated

=> Selected Limit

3-14

WHC-SD-WM-DP-115, REV. 1  
WHC-SD-WM-DP-115, REV. 0

Summary Tables - Preliminary Safety Screening Results  
C-204

CORE NUMBER: n/a  
SEGMENT #: 95-AUG-023

TABLE 6

SEGMENT PORTION: W Whole Segment

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000890			% Water by TGA using Mettler	%	17.000	110.000	99.76	n/a	59.92	56.08	58.00	6.62	n/a	n/a	n/a
S95T000890			DSC Exotherm Dry Calculated	Joules/g Dry	-999.000	481.010	n/a	n/a	952.1	665.7	808.9	35.4	n/a	1.00e-04	n/a
S95T000890			DSC Exotherm using Mettler	Joules/g	-999.000	481.010	107.2	n/a	399.9	279.6	339.8	35.4	n/a	n/a	n/a
S95T000891	F		Alpha of Digested Solid	uCi/g	-999.000	41.010	105.7	1.40e-02	5.11e-02	5.19e-2	5.15e-02	1.55	n/a	7.00e-03	17.6

=> Limit violated

=> Selected Limit

3-15

WHC-SD-WM-DP-115, REV. 0

WHC-SD-WM-DP-115, REV. 1

**WHC-SD-WM-DP-115, REV. 1**

**SAMPLE ANALYSES RESULTS**

WHC-SD-WM-DP-115, REV. 1

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# LABCORE Data Entry Template for Worklist# 1378

Analyst: SMF Instrument: DSC0 1 Book # 12N14-A

Method: LA-514-113 Rev/Mod B-1

WHC-SD-WM-DP- 115, REV. 1

Worklist Comment: Please run C-204 DSC under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID	<u>28.45</u>	<u>30.7</u>	<u>N/A</u>	Joules/g
95000069	C-204	2 SAMPLE	S95T000878	0	DSC-01	SOLID	<u>N/A</u>	<u>&gt;195.7</u>		Joules/g
95000069	C-204	3 DUP	S95T000878	0	DSC-01	SOLID	<u>&gt;195.7</u>	<u>&gt;542.0</u>	<u>N/A</u>	Joules/g
95000069	C-204	4 TRIPL	S95T000878	0	DSC-01	SOLID	<u>&gt;195.7</u>	<u>&gt;305.9</u>	<u>N/A</u>	Joules/g
		5 STD			DSC-01	SOLID	<u>28.45</u>	<u>29.5</u>	<u>N/A</u>	Joules/g
95000069	C-204	6 SAMPLE	S95T000881	0	DSC-01	SOLID	<u>N/A</u>	<u>&gt;286.7</u>		Joules/g
95000069	C-204	7 DUP	S95T000881	0	DSC-01	SOLID	<u>&gt;286.7</u>	<u>&gt;33.4</u>	<u>N/A</u>	Joules/g
95000069	C-204	8 TRIPL	S95T000881	0	DSC-01	SOLID	<u>&gt;286.7</u>	<u>&gt;508.8</u>	<u>N/A</u>	Joules/g

## Final page for worklist # 1378

See attached for signatures 5/18/95  
Analyst Signature BDV Date  
Verified by Blandina Valenzuela 5/22/95

[Signature] 5-18-95  
Analyst Signature Date

The exotherm values reported are a not the total energy produced from the reaction, the thermogram never returned back to the baseline. Therefore, the results should be considered greater than the reported values.

Data Entry Comments: S95T000878 produced one endotherm of 551.4 J/g at 106.8°C.  
S95T000881 produced one endotherm of 484.4 J/g at 134.6°C

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

# LABCORE Data Entry Template for Worklist# 1378

Analyst: SMF Instrument: DSC0 Book # 12N14-A

Method: LA-514-113 Rev/Mod B-1 WHC-SD-WM-DP- 115, REV. 1

Worklist Comment: Please run C-204 DSC under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID			N/A	Joules/g
95000069	C-204	2 SAMPLE	S95T000878	0	DSC-01	SOLID	N/A			Joules/g
95000069	C-204	3 DUP	S95T000878	0	DSC-01	SOLID			N/A	Joules/g
95000069	C-204	4 SAMPLE	S95T000881	0	DSC-01	SOLID	N/A			Joules/g
95000069	C-204	5 DUP	S95T000881	0	DSC-01	SOLID			N/A	Joules/g

Final page for worklist # 1378

Smfulton 5-17-95  
Analyst Signature Date

\_\_\_\_\_  
Analyst Signature Date

A triplicate was run on both samples.  
5/18/95  
BDV

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number,  
R = Replicate Number, A = Aliquot Code.

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SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT  
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 3-20 TO 3-24.

DSC STD 12N14-A

6.580 mg

Rate: 10.0 °C/min

File: 00015.001

Ident: 0.0

DSC METTLER 16-May-95

222-S Laboratory

exo >

10. mW

Integration

Delta H 202 mJ

30.7 J/g

Peak 158.9 °C

-13.4 mW

120.

140.

160.

180. °C

Blandina Valenzuela for SM Fulton  
5/17/95

3-20

WHC-SD-WM-DP-115, REV. 1

WHC-SD-WM-DP-115, REV. 0

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S95T000878 N2

22.047 mg

Rate: 10.0 °C/min

File: 00017.001 DSC METTLER 16-May-95

Ident: 0.0

222-S Laboratory

exo >

Integration

Delta H 12157 mJ

551.4 J/g

Peak 106.8 °C

-11.5 mW

Integration

Delta H 4315 mJ

195.7 J/g

Peak 448.6 °C

5.8 mW

Integration

Delta H 30 mJ

1.4 J/g

Peak 289.4 °C

-0.6 mW

10. mW

100.

200.

300.

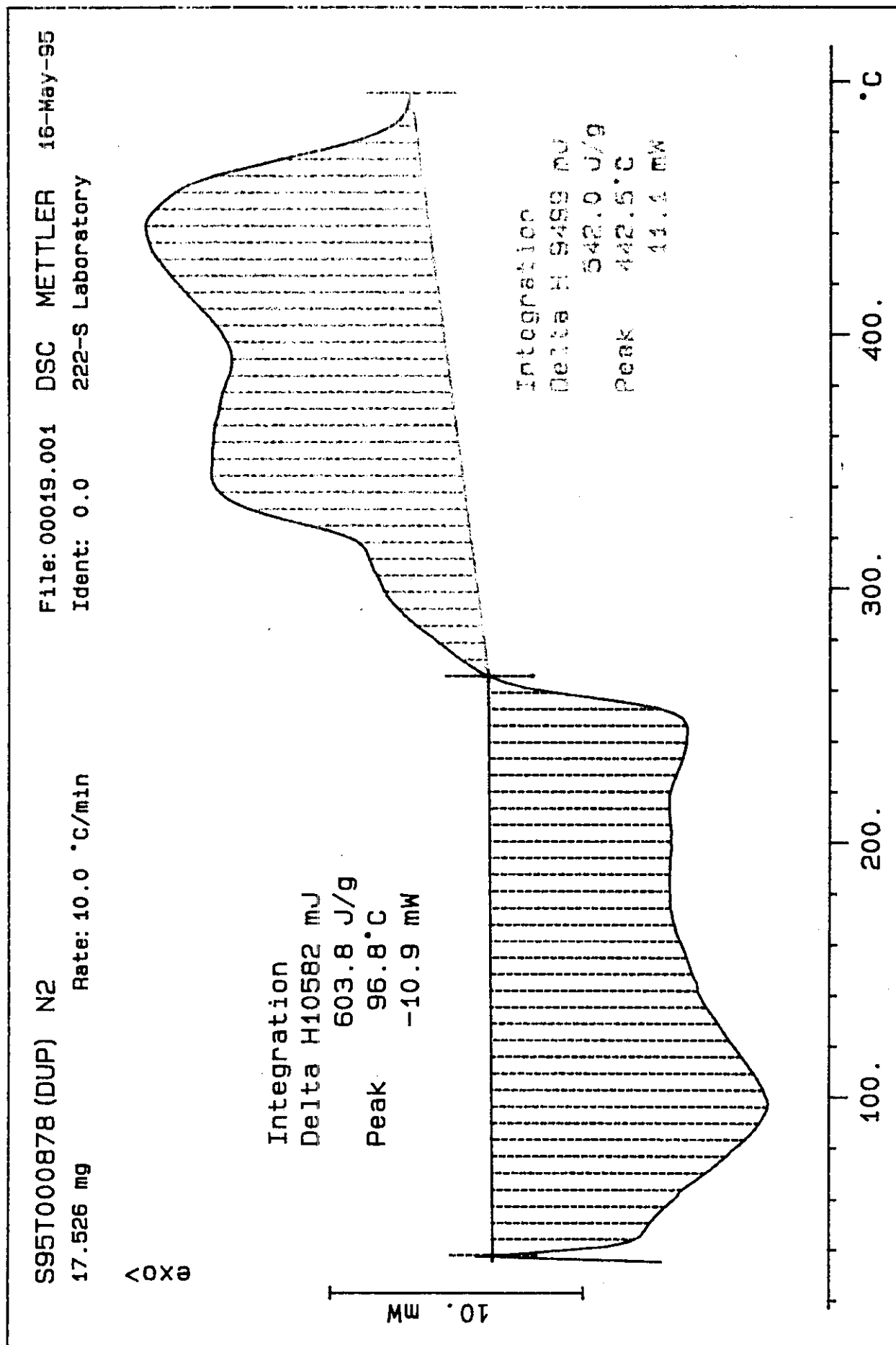
400.

°C

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WHC-SD-WM-DP-112, REV. 1  
WHC-SD-WM-DP-115, REV. 0

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S95T000878 (TRIPL) N2  
35.625 mg Rate: 10.0 °C/min

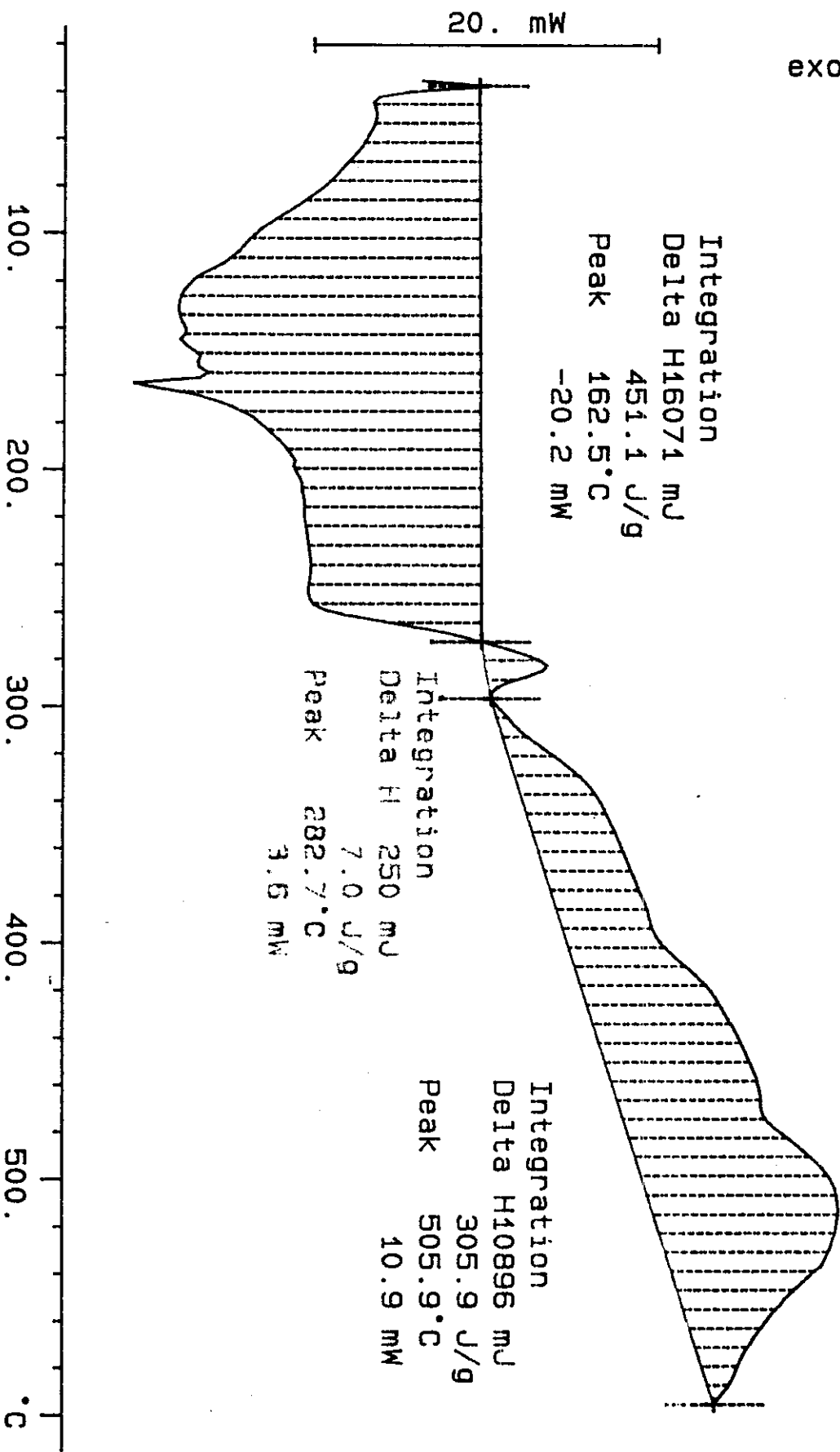
File: 00021.001 DSC METTLER 16-May-95  
Ident: 0.0 222-S Laboratory

exo>

Integration  
Delta H16071 mJ  
451.1 J/g  
Peak 162.5°C  
-20.2 mW

Integration  
Delta H 250 mJ  
7.0 J/g  
Peak 282.7°C  
3.6 mW

Integration  
Delta H10896 mJ  
305.9 J/g  
Peak 505.9°C  
10.9 mW



3-23

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DSC STD 12N14-A

6.746 mg

Rate: 10.0 °C/min

File: 00036.001

Ident: 0.0

DSC METTLER

17-May-95

222-S Laboratory

exo

10. mW

Integration

Delta H 199 mJ

29.5 J/g

Peak 158.7°C

-13.5 mW

120.

140.

160.

180. °C

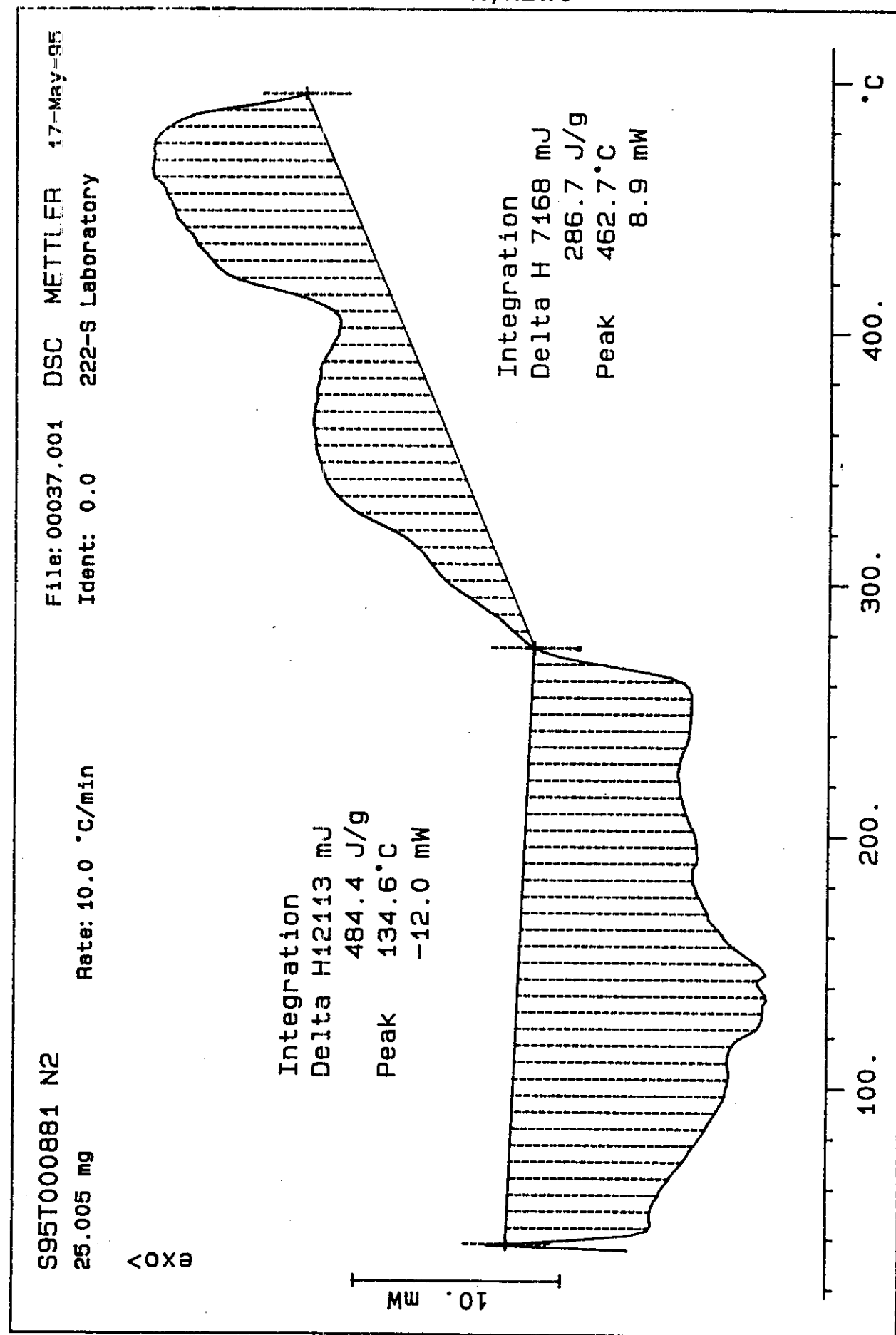
*Blandina Valenzuela for SM Fulton 5/17/95*

3-24

WHC-SD-WM-DP-115, REV. 1

WHC-SD-WM-DP-115, REV. 0

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S95T000881 (DUP) N2

25.525 mg

Rate: 10.0 °C/min

File: 00039.001

Ident: 0.0

DSC METTLER

17-May-95

222-S Laboratory

exo>

Integration

Delta H 14902 mJ

583.8 J/g

Peak 102.8 °C

-11.8 mW

10. mW

Integration

Delta H 853 mJ

33.4 J/g

Peak 341.6 °C

1.7 mW

100.

200.

300.

400.

°C

3-26

WHC-SD-WM-DP-112, REV. 1  
WHC-SD-WM-DP-115, REV. 0

BEST AVAILABLE COPY

S95T000881 (TRIPL) N2  
26.737 mg

Rate: 10.0 °C/min

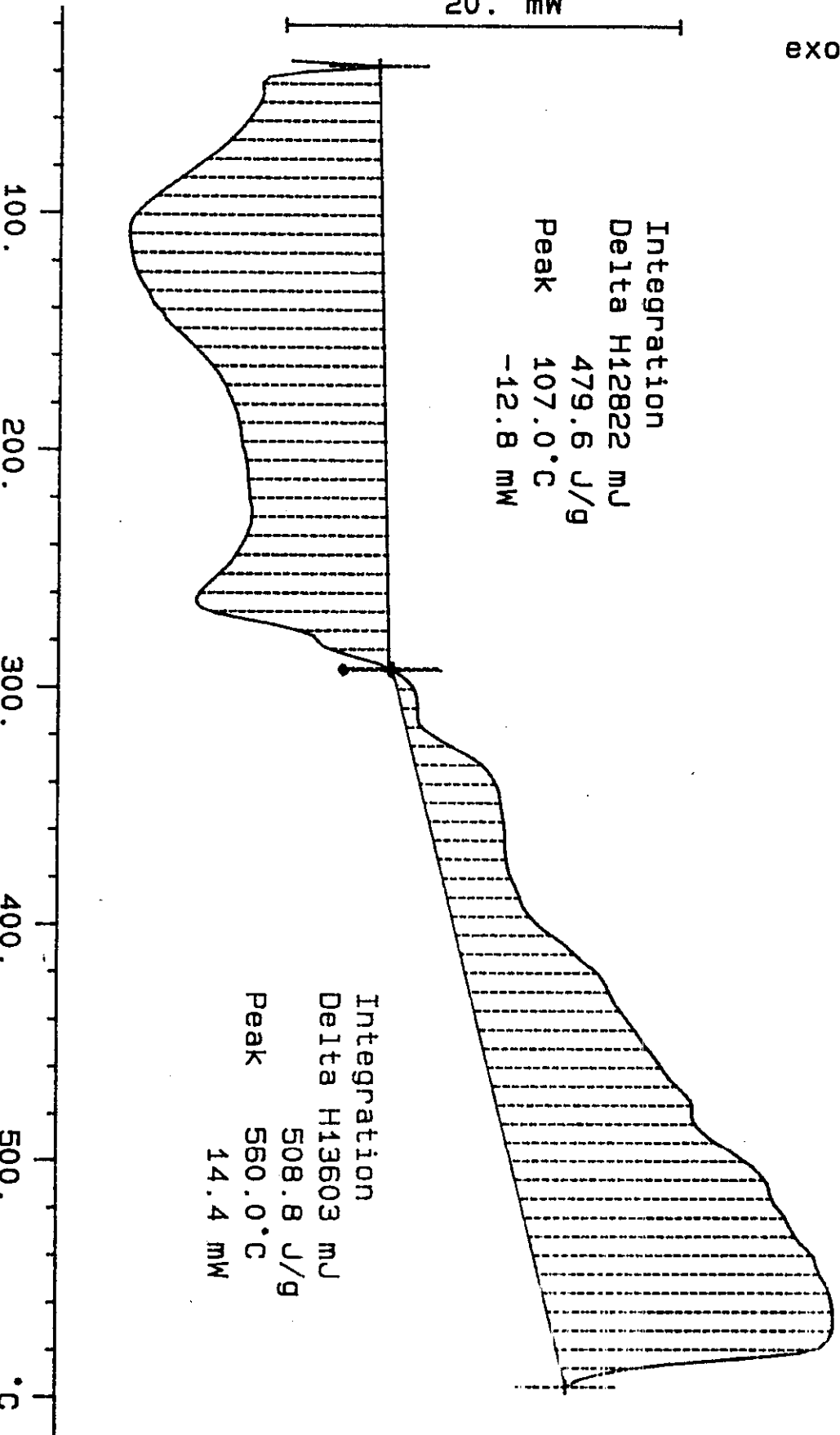
File: 00041.001 DSC METTLER 17-May-95  
Ident: 0.0 222-S Laboratory

exo>

Integration  
Delta H12822 mJ  
479.6 J/g  
Peak 107.0 °C  
-12.8 mW

Integration  
Delta H13603 mJ  
508.8 J/g  
Peak 560.0 °C  
14.4 mW

20. mW



3-27

# LABCORE Data Entry Template for Worklist# 1379

Analyst: SMF Instrument: DSC0 1 Book # 12N14-A

Method: LA-514-113 Rev/Mod B-1 WHC-SD-WM-DP- 115, REV. 1

Worklist Comment: Please run C-204 DSC under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID	<u>28.45</u>	<u>30.5</u>	<u>N/A</u>	Joules/g
95000069	C-204	2 SAMPLE	S95T000890	0	DSC-01	SOLID	<u>N/A</u>	<u>399.9</u>		Joules/g
95000069	C-204	3 DUP	S95T000890	0	DSC-01	SOLID	<u>399.9</u>	<u>279.6</u>	<u>N/A</u>	Joules/g
95000069	C-204	4 TRIPL	S95T000890	0	DSC-01	SOLID	<u>399.9</u>	<u>345.6</u>	<u>N/A</u>	Joules/g

## Final page for worklist # 1379

See attached for signatures 5/11/95  
Analyst Signature                      Date                       
BDV

                      
Analyst Signature                      Date 5-12-95

Verified 5/12/95 James M. Foy

Data Entry Comments: Sample produced one endotherm at 132.6°C with a  
delta H of 422.2 J/g. Sample looked like stiff dark chocolate frosting.

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number,  
R = Replicate Number, A = Aliquot Code.

# LABCORE Data Entry Template for Worklist# 1379

Analyst: SMF Instrument: DSC0 Book # 12N14-7

Method: LA-514-113 Rev/Mod B-1 WHC-SD-WM-DP-115, REV. 1

Worklist Comment: Please run C-204 DSC under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID			N/A	Joules/g
95000069	C-204	2 SAMPLE	S95T000890	0	DSC-01	SOLID	N/A			Joules/g
95000069	C-204	3 DUP	S95T000890	0	DSC-01	SOLID			N/A	Joules/g

Final page for worklist # 1379

SMF  
Analyst Signature Date

Analyst Signature Date

Triplicate was run. 5/11/95  
BDV

Data Entry Comments:

sample is like stiff dark chocolate frosting

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT  
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 330 TO 333.

DSC STD 12N14-A

6.632 mg

Rate: 10.0 °C/min

File: 00033.001

DSC METTLER 10-May-95

Ident: 0.0

222-S Laboratory

exo

10. mW

Integration  
Delta H 202 mJ  
30.5 J/g  
Peak 158.6 °C  
-13.5 mW

120.

140.

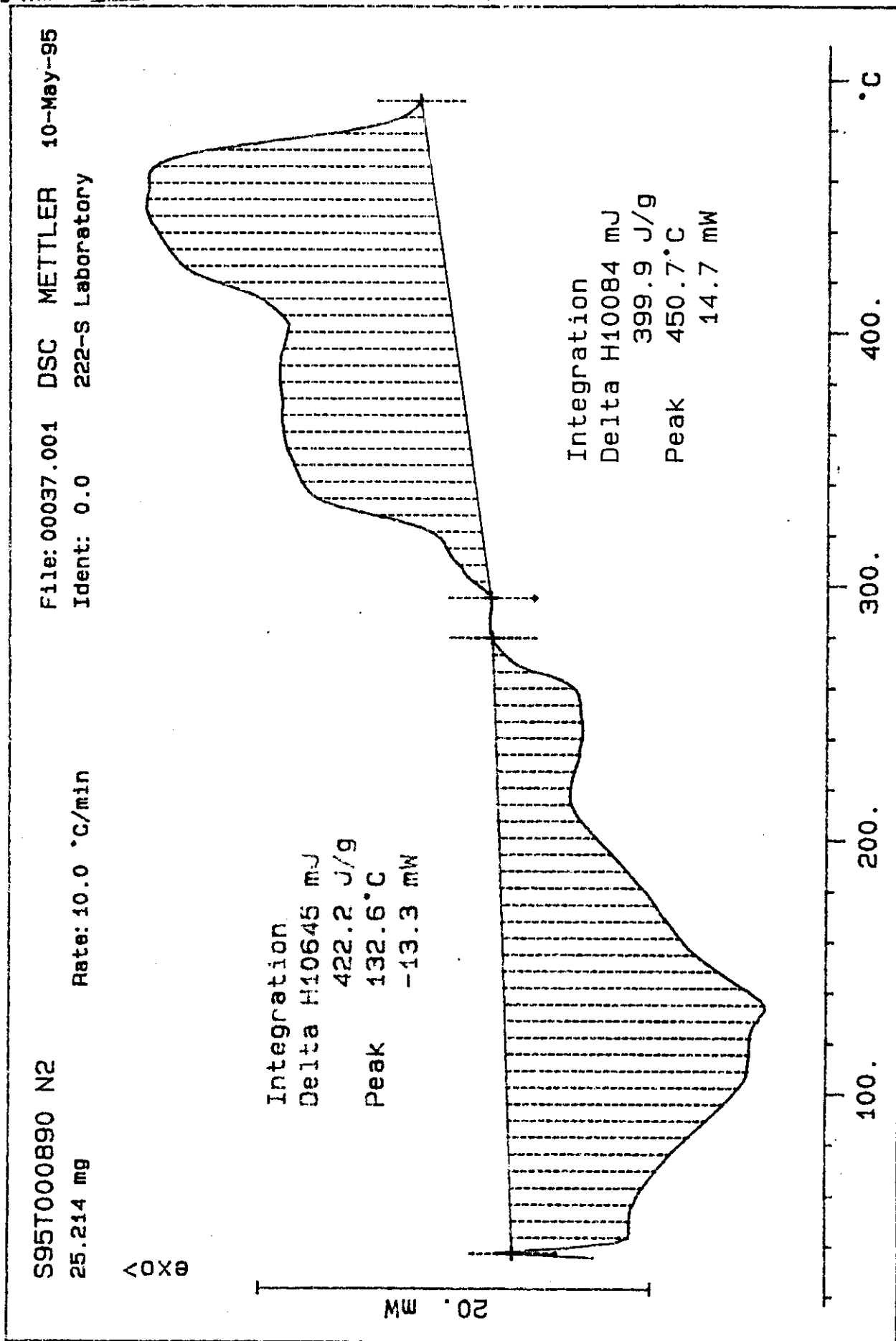
160.

180.

°C

*Susie M. Dulter* 5-10-95

3-30



S95T000890 (DUP) N2  
20.209 mg  
File: 00039.001 DSC METTLER 10-May-95  
Ident: 0.0 222-S Laboratory

Rate: 10.0 °C/min

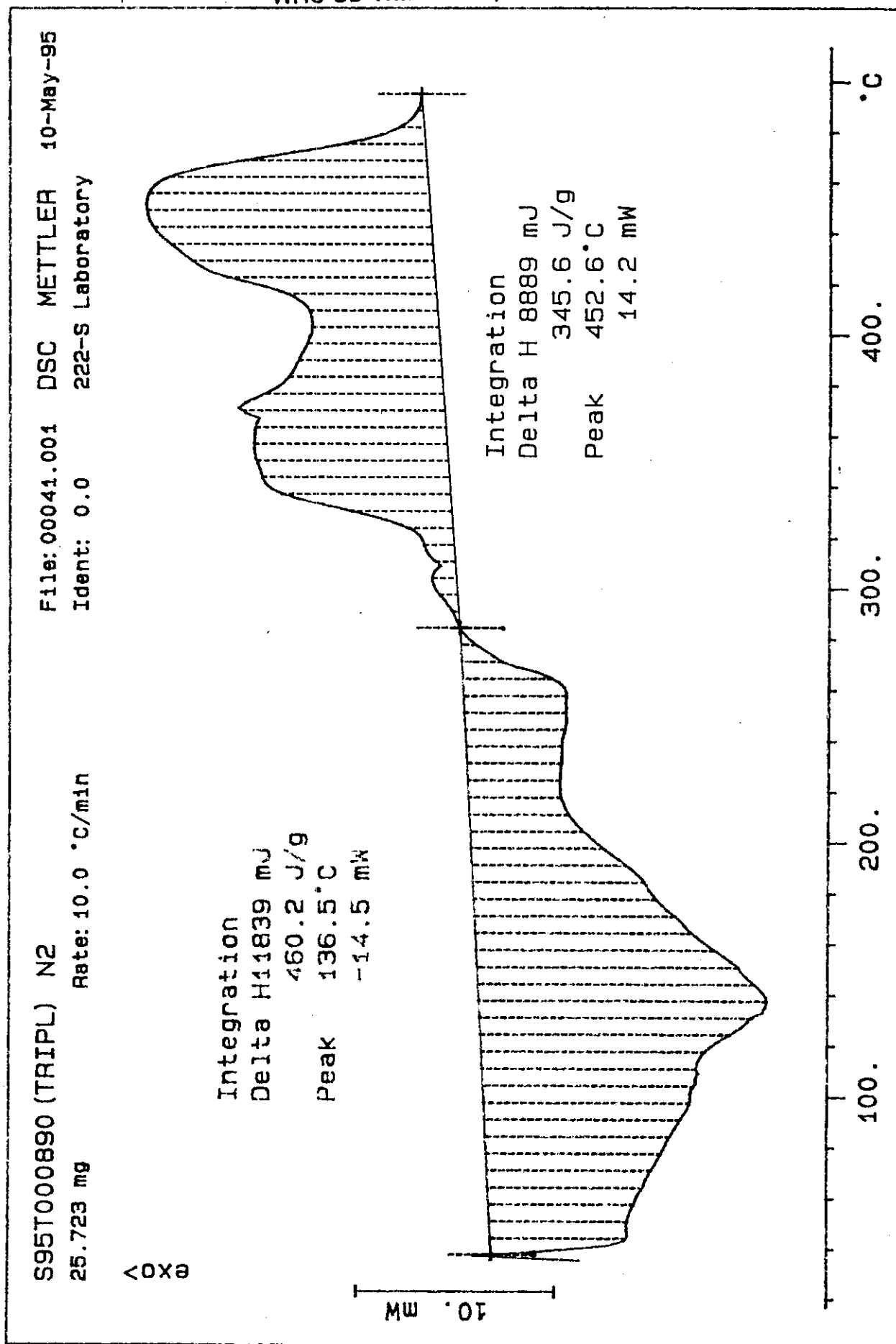
EXOV

Integration  
Delta H10851 mJ  
536.9 J/g  
Peak 223.0 °C  
-9.8 mW

Integration  
Delta H 5651 mJ  
279.6 J/g  
Peak 336.6 °C  
12.1 mW

10. mW

100. 200. 300. 400. °C





# LABCORE Data Entry Template for Worklist# 1431

Analyst: BDV Instrument: DSC0 2 Book # —

Method: LA-514-113 Rev/Mod —

WHC-SD-WM-DP-115, REV. 1

Worklist Comment: Calculated dry DSC for C-204. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
95000069	C-204	1 SAMPLE	S95T000878	0	DSC-02	SOLID	N/A	>445.6		Joules/g Dry
95000069	C-204	2 DUP	S95T000878	0	DSC-02	SOLID	>445.6	>1234.0	N/A	Joules/g Dry
95000069	C-204	3 TRIPL	S95T000878	0	DSC-02	SOLID	>445.6	>696.5	N/A	Joules/g Dry
95000069	C-204	4 SAMPLE	S95T000881	0	DSC-02	SOLID	N/A	>647.3		Joules/g Dry
95000069	C-204	5 DUP	S95T000881	0	DSC-02	SOLID	>647.3	>76.1	N/A	Joules/g Dry
95000069	C-204	6 TRIPL	S95T000881	0	DSC-02	SOLID	>647.3	>1149.0	N/A	Joules/g Dry
95000069	C-204	7 SAMPLE	S95T000890	0	DSC-02	SOLID	N/A	952.1		Joules/g Dry
95000069	C-204	8 DUP	S95T000890	0	DSC-02	SOLID	952.1	665.7	N/A	Joules/g Dry
95000069	C-204	9 TRIPL	S95T000890	0	DSC-02	SOLID	952.1	822.9	N/A	Joules/g Dry

Data entered & verified by **Final page for worklist # 1431**

Blandina Valenzuela 5/22/95

Analyst Signature

Date

Analyst Signature

Date

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.



## WHC-SD-WM-DP-115, REV. 0

The following DSC runs were performed in an attempt to better understand the exothermic characteristics of the C-204 samples. The samples were pre-dried before being run on the DSC. These are unofficial results. These test runs are described in the narrative.

WHC-SD-WM-DP-115, REV. 1

Sample S95T000878	TEST
Sample S95T000881	TEST

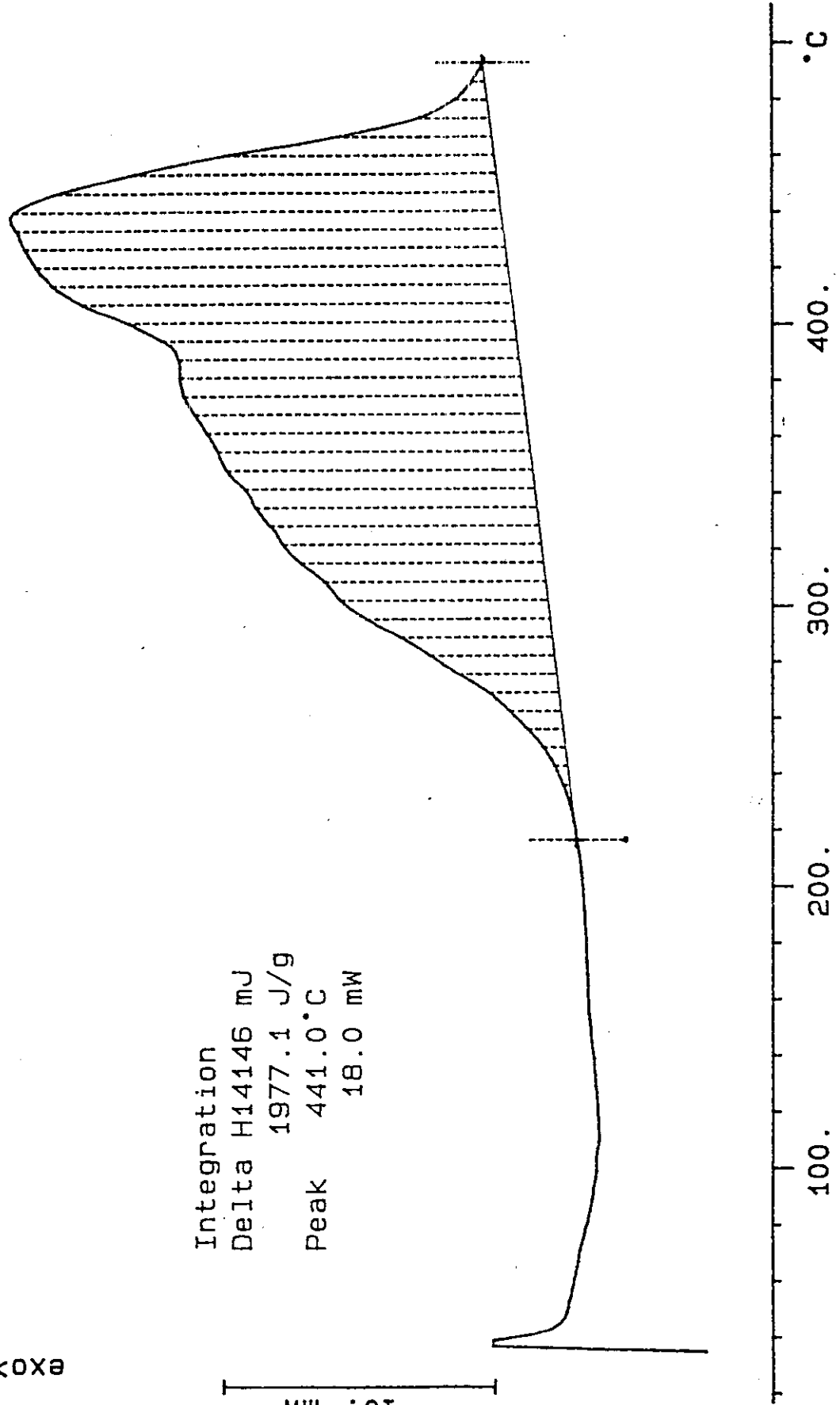
COPY

S95T000878 (TEST) N2  
7.155 mg  
Rate: 10.0 °C/min  
File: 00055.001 DSC METTLER 19-May-95  
Ident: 0.0 222-S Laboratory

EXOV

Integration  
Delta H14146 mJ  
1977.1 J/g  
Peak 441.0 °C  
18.0 mW

10. mW



GOPY

S95T000881 (TEST) N2  
12.911 mg

Rate: 10.0 °C/min

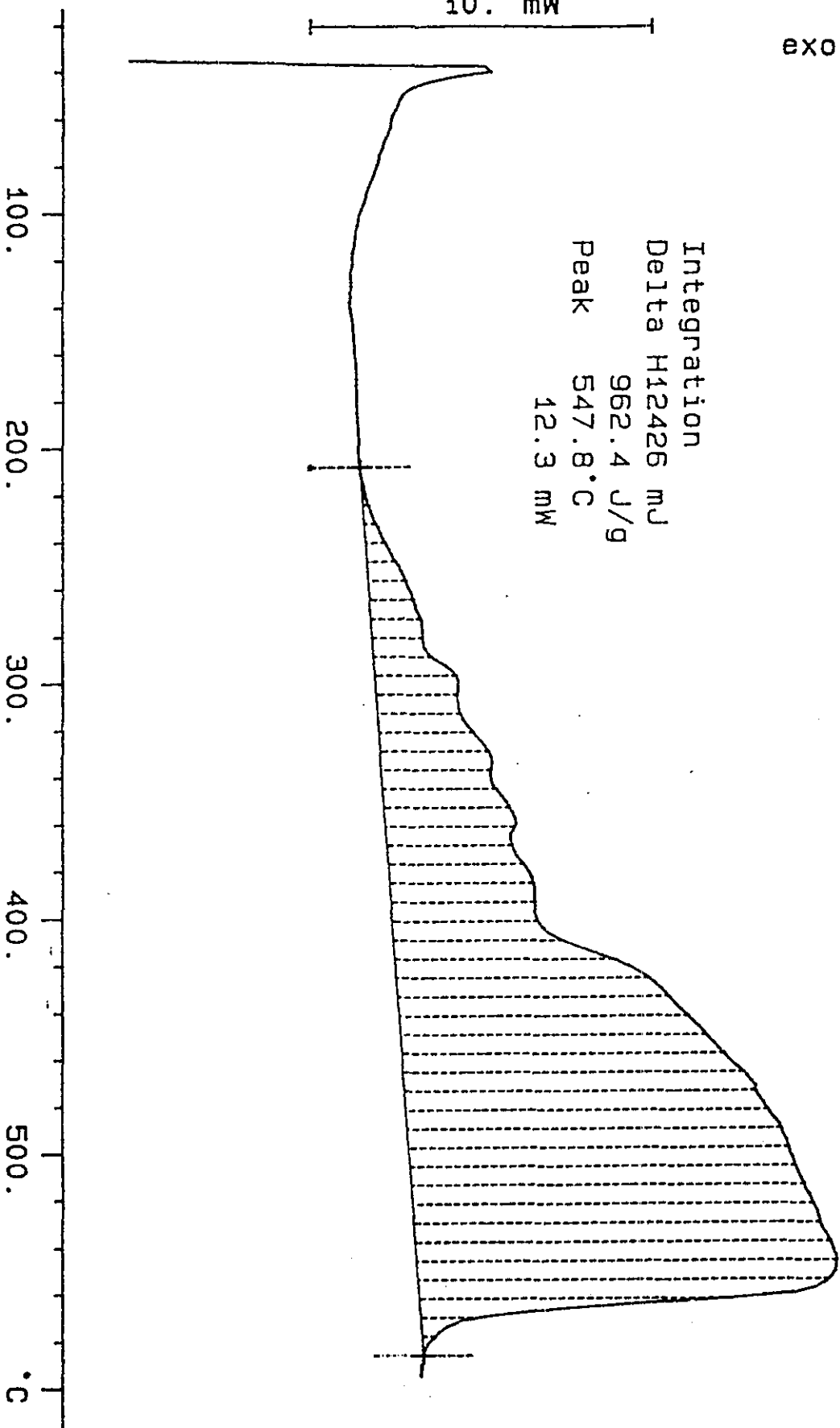
File: 00057.001 DSC METTLER 19-May-95  
Ident: 0.0 222-S Laboratory

exo>

3-38

10. mW

Integration  
Delta H12426 mJ  
962.4 J/g  
Peak 547.8 °C  
12.3 mW



# LABCORE Data Entry Template for Worklist# 1374

Analyst: SMF Instrument: TGA0 1 Book # 42N8-A

Method: LA-560-112 Rev/Mod A-2

Worklist Comment: Please run C-204 TGA under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			TGA-01	SOLID	<u>59.19</u>	<u>59.39</u>	<u>N/A</u>	%
95000069	C-204	2 SAMPLE	S95T000878	0	TGA-01	SOLID	<u>N/A</u>	<u>58.32</u>		%
95000069	C-204	3 DUP	S95T000878	0	TGA-01	SOLID	<u>58.32</u> <u>5/22/95</u> <u>50.44</u>	<u>50.44</u>	<u>N/A</u>	%
95000069	C-204	4 TRIPL	S95T000878	0	TGA-01	SOLID	<u>58.32</u>	<u>59.48</u>	<u>N/A</u>	%
		5 STD			TGA-01	SOLID	<u>59.19</u>	<u>58.77</u>	<u>N/A</u>	%
95000069	C-204	6 SAMPLE	S95T000881	0	TGA-01	SOLID	<u>N/A</u>	<u>55.02</u>		%
95000069	C-204	7 DUP	S95T000881	0	TGA-01	SOLID	<u>55.02</u>	<u>56.39</u>	<u>N/A</u>	%

Final page for worklist # 1374

See attached for signatures  
Analyst Signature                      Date                     

                     5-17-95  
Analyst Signature                      Date                     

Verified by Blandina Valenzuela 5/22/95

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

# LABCORE Data Entry Template for Worklist# 1374

Analyst: SMF Instrument: TGA0 \_\_\_\_\_ Book # 42 N8-A

Method: LA-560-112 Rev/Mod A-2 WHC-SD-WM-DP-115, REV. 1

Worklist Comment: Please run C-204 TGA under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			TGA-01	SOLID	_____	_____	N/A	%
95000069	C-204	2 SAMPLE	S95T000878	0	TGA-01	SOLID	N/A	_____	_____	%
95000069	C-204	3 DUP	S95T000878	0	TGA-01	SOLID	_____	_____	N/A	%
95000069	C-204	4 SAMPLE	S95T000881	0	TGA-01	SOLID	N/A	_____	_____	%
95000069	C-204	5 DUP	S95T000881	0	TGA-01	SOLID	_____	_____	N/A	%

Final page for worklist # 1374

Sm-fulton 5-17-95  
Analyst Signature Date

\_\_\_\_\_  
Analyst Signature Date

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT  
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 3-41 TO 3-47.

TGA STD 42N8-A

18.284 mg

Rate: 10.0 °C/min

File: 00016.001

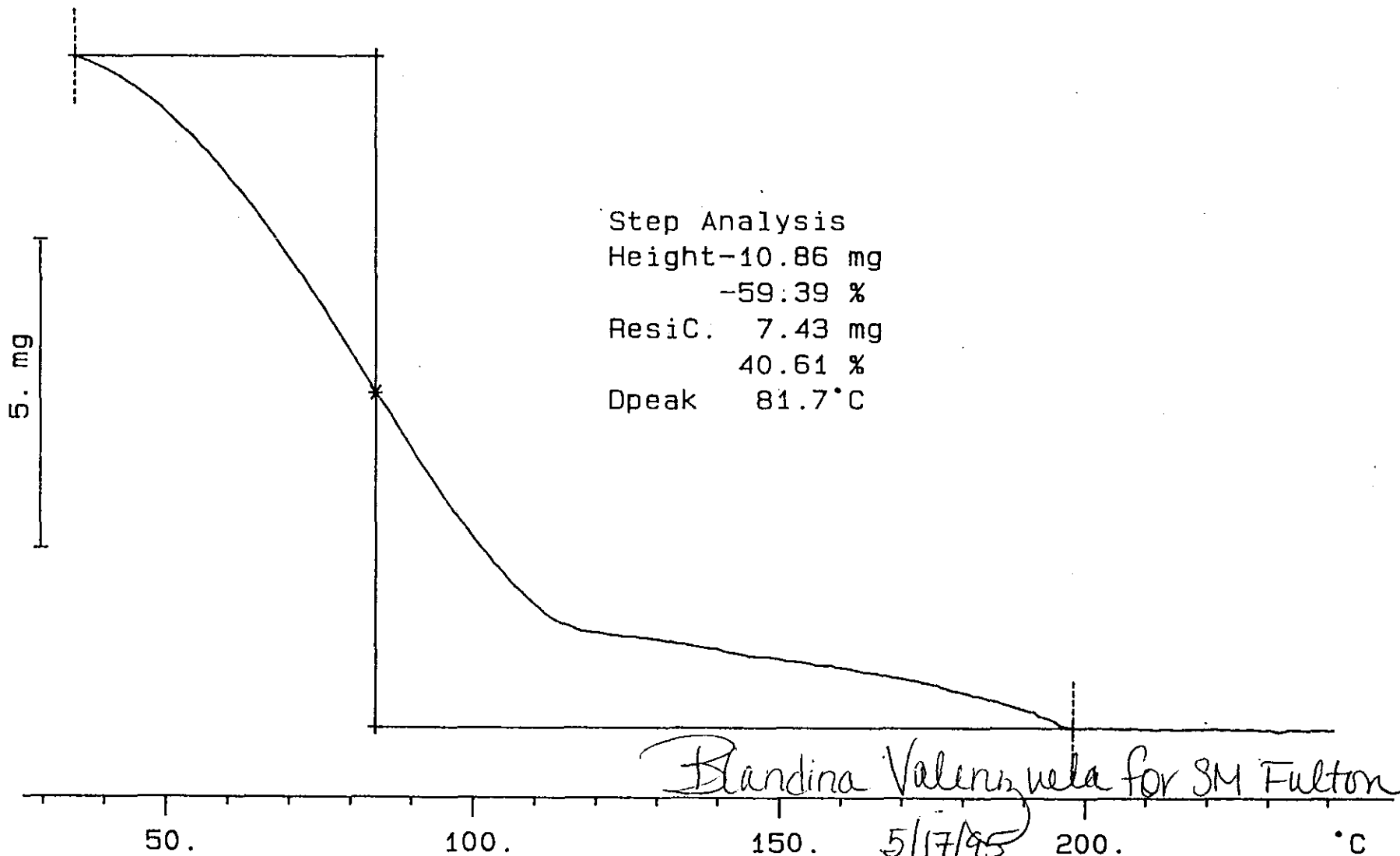
TG

METTLER

16-May-95

Ident: 0.0

222-S Laboratory

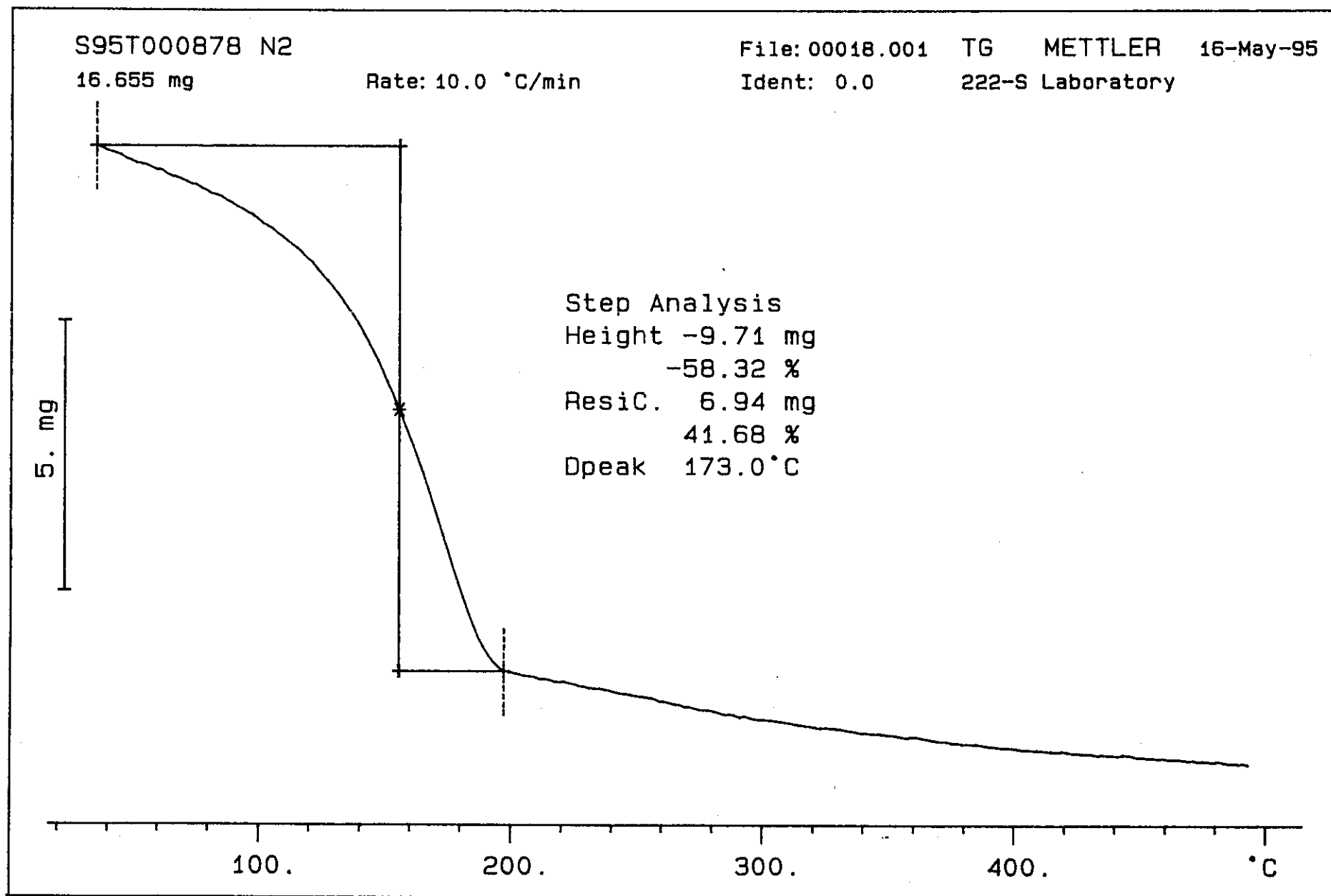


3-41

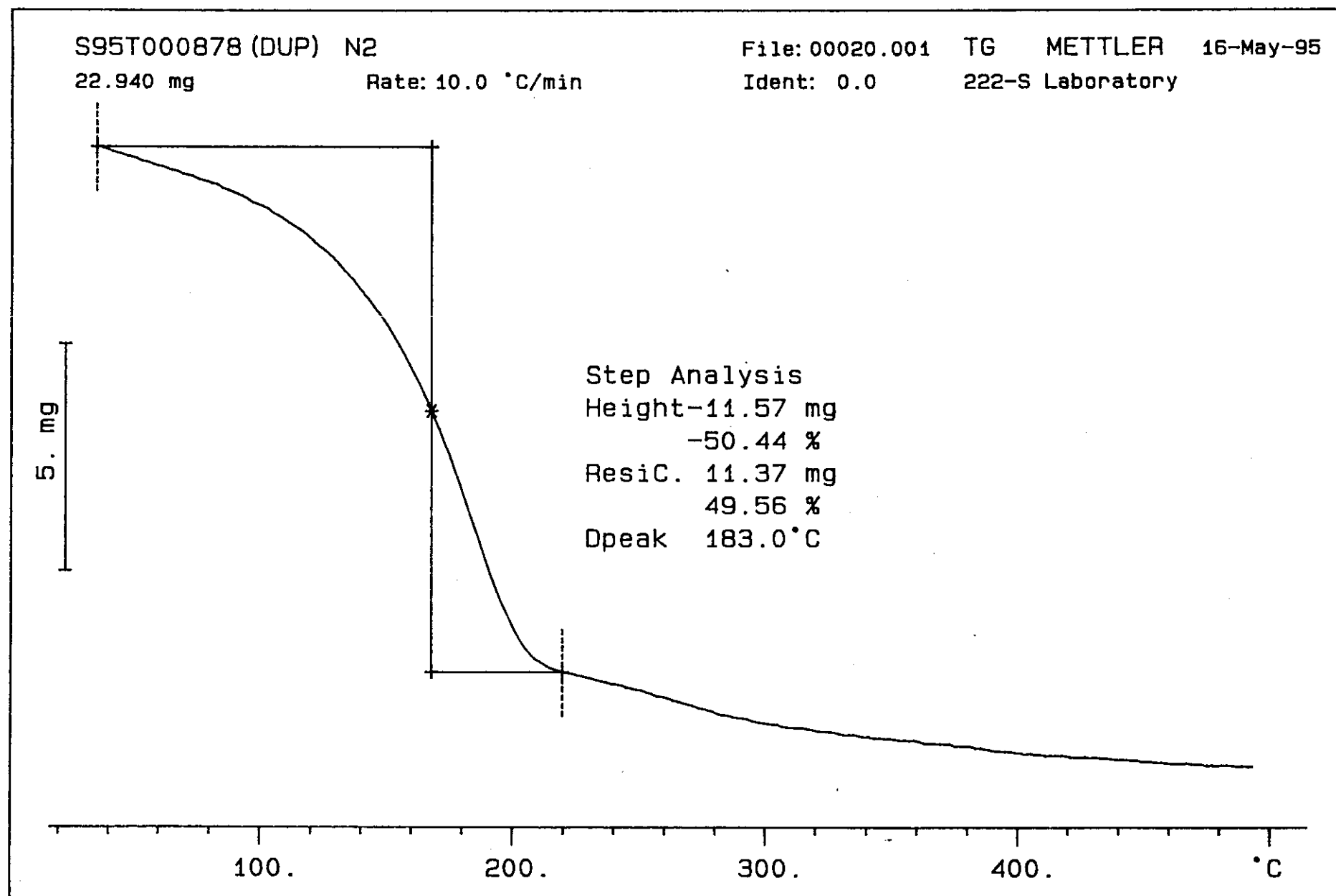
WHC-SD-WM-DP-115, REV.0



3-42



3-43



S95T000878 (TRIPL) N2

10.260 mg

Rate: 10.0 °C/min

File: 00022.001

TG

METTLER

16-May-95

Ident: 0.0

222-S Laboratory

Step Analysis

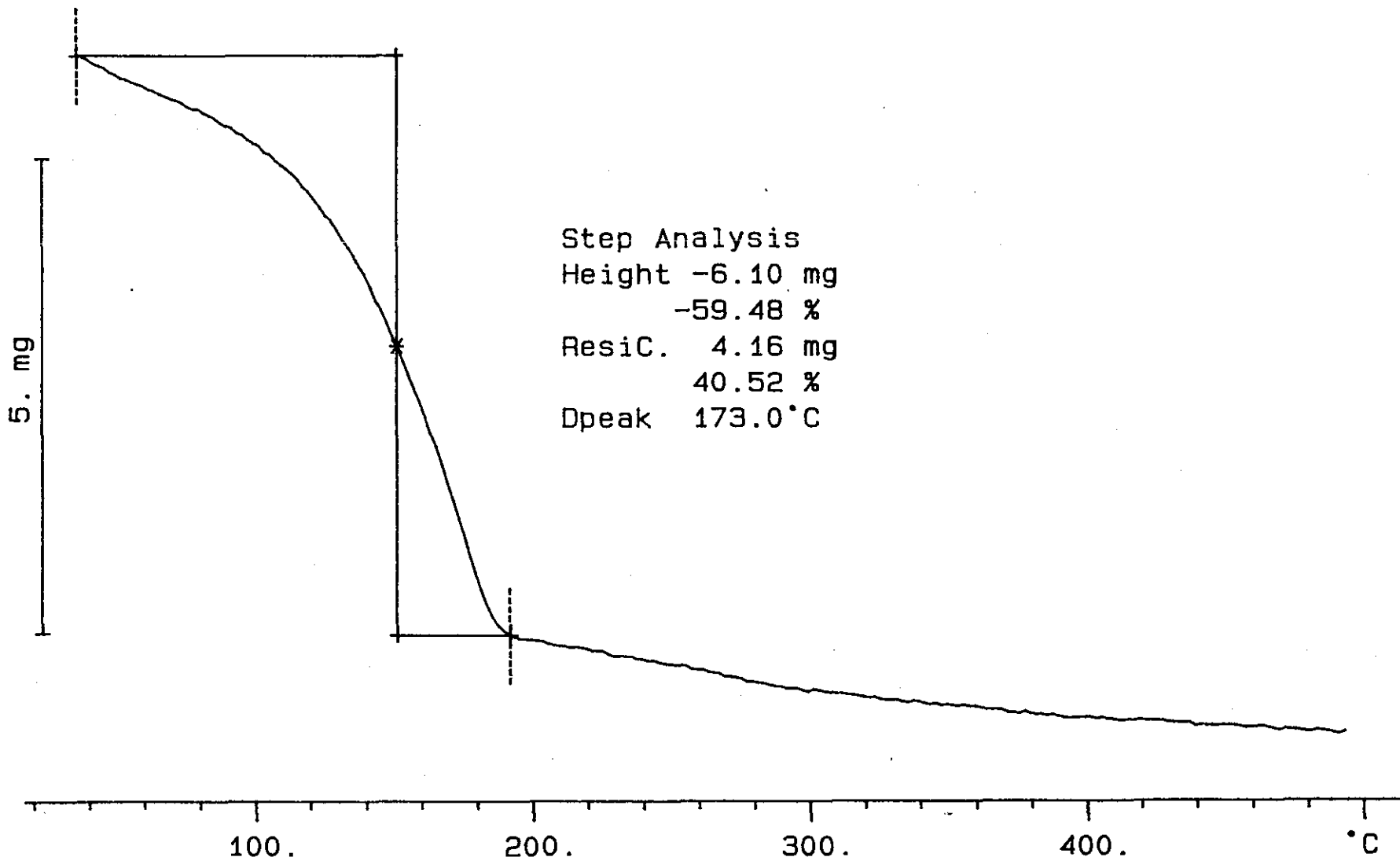
Height -6.10 mg

-59.48 %

ResiC. 4.16 mg

40.52 %

Dpeak 173.0 °C



TGA STD 42N8-A

18.250 mg

Rate: 10.0 °C/min

File: 00035.001

Ident: 0.0

TG

METTLER

17-May-95

222-S Laboratory

Step Analysis

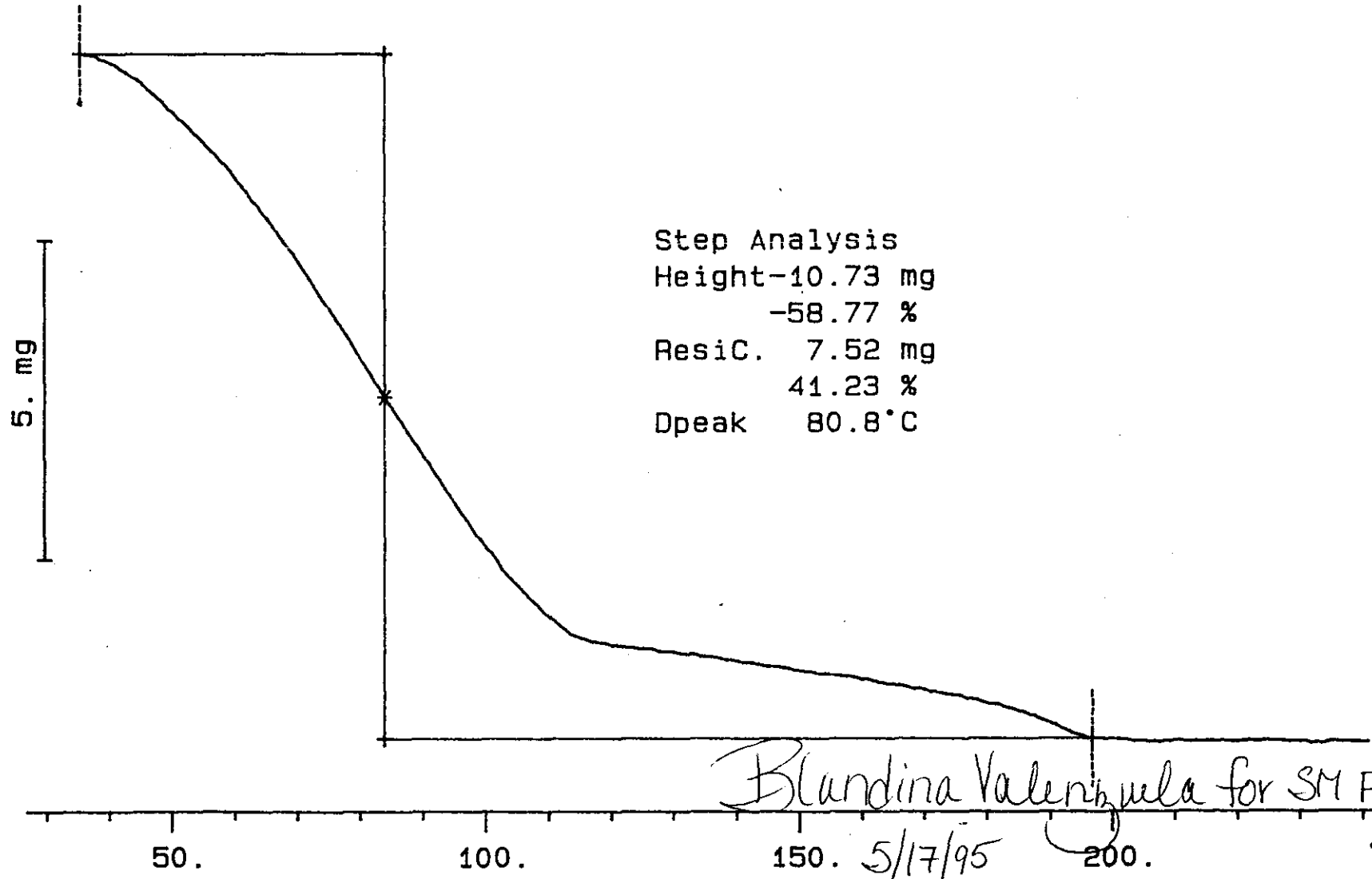
Height-10.73 mg

-58.77 %

ResiC. 7.52 mg

41.23 %

Dpeak 80.8 °C



3-45

S95T000881 N2

23.401 mg

Rate: 10.0 °C/min

File: 00038.001

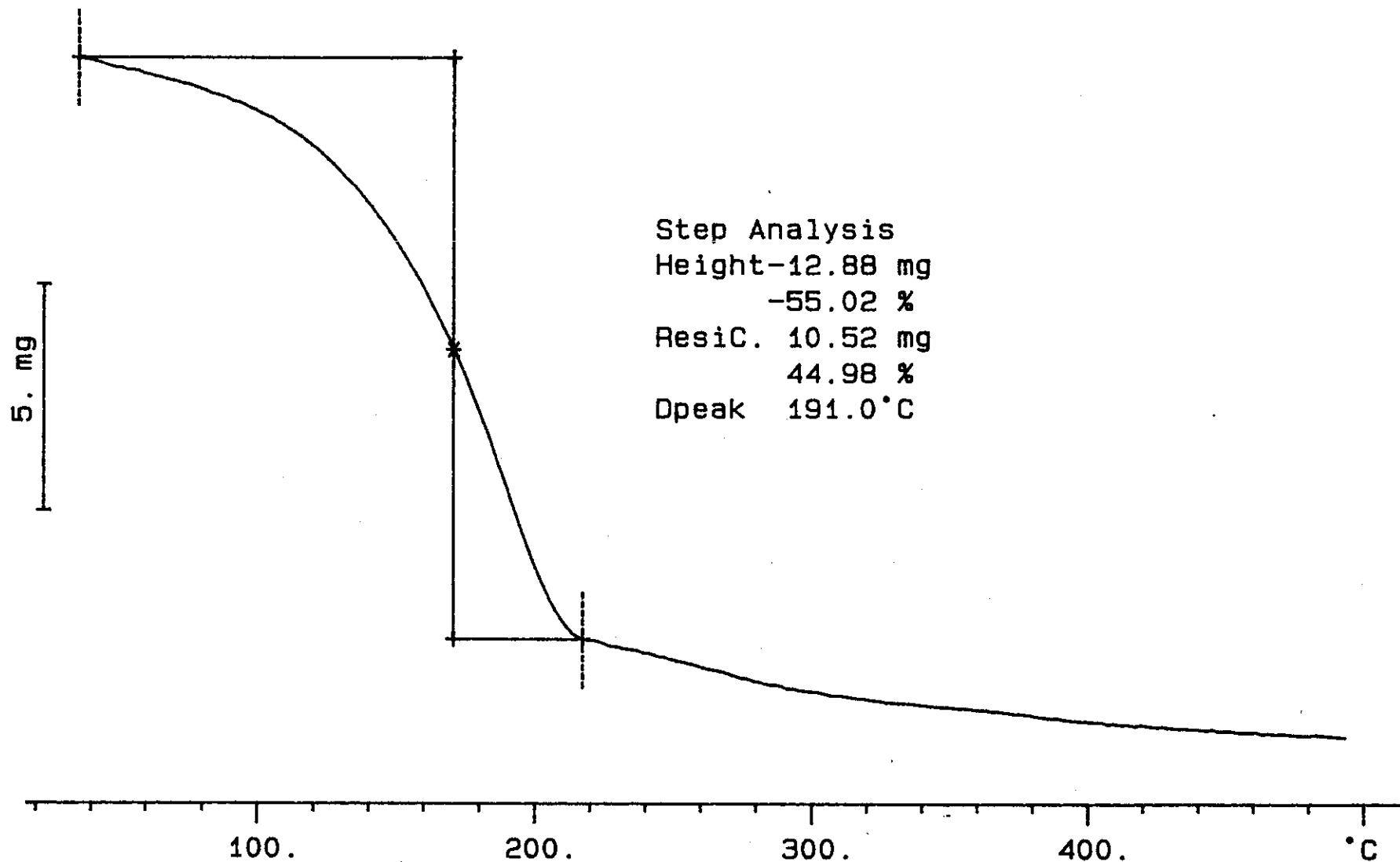
TG

METTLER

17-May-95

Ident: 0.0

222-S Laboratory



3-47

S95T000881 (DUP) N2

19.399 mg

Rate: 10.0 °C/min

File: 00040.001

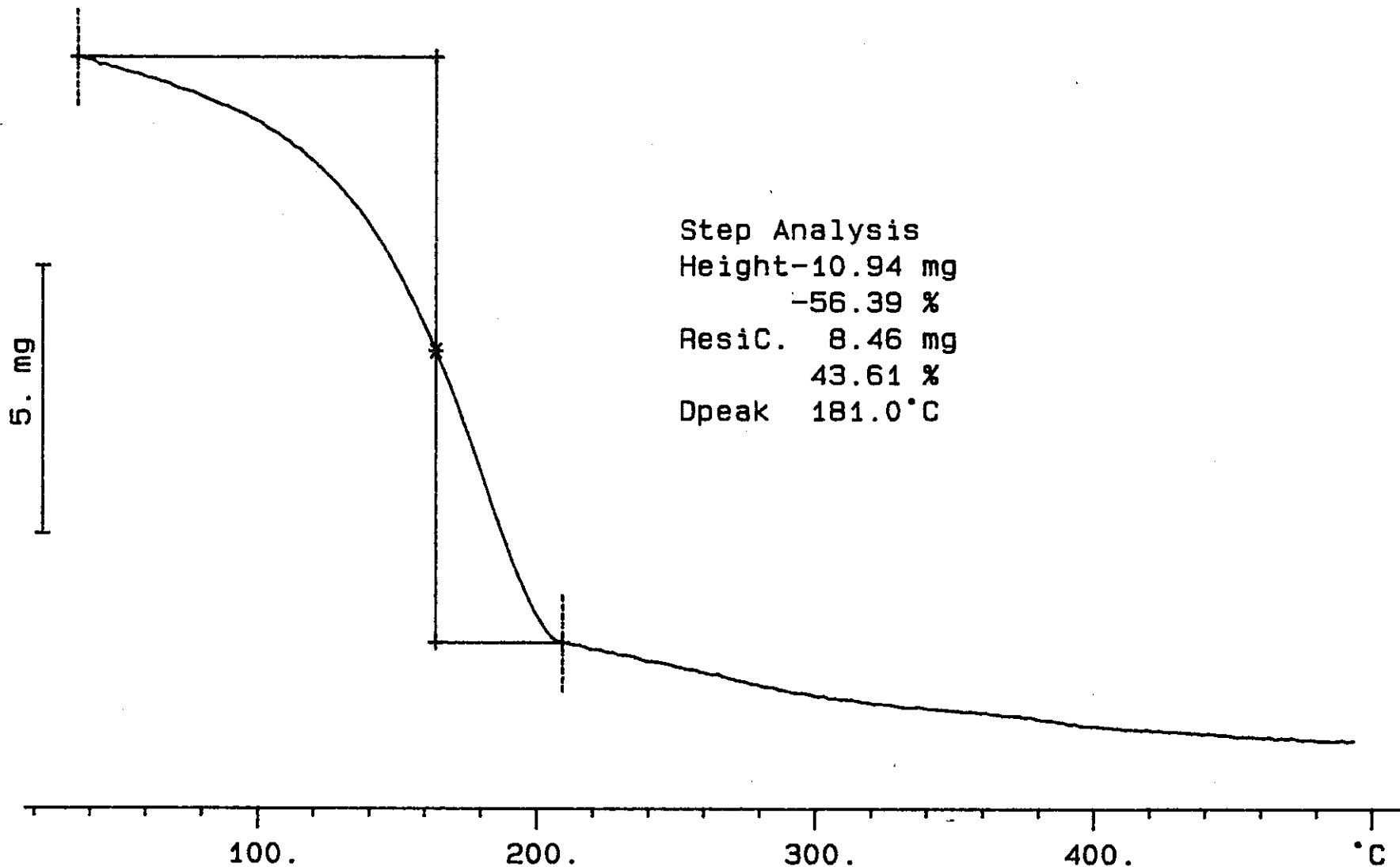
TG

METTLER

17-May-95

Ident: 0.0

222-S Laboratory



WHC-SD-WM-DP-115, REV. 1

WHC-SD-WM-DP-115, REV. 0

# LABCORE Data Entry Template for Worklist# 1375

Analyst: SMF Instrument: TGA0 1 Book # 42N8-A

Method: LA-560-112 Rev/Mod A-2 WHC-SD-WM-DP-115, REV. 1

Worklist Comment: Please run C-204 TGA under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			TGA-01	SOLID	<u>59.19</u>	<u>59.05</u> <u>59.04</u>	<u>5/11/95</u> <u>N/A</u>	%
95000069	C-204	2 SAMPLE	S95T000890	0	TGA-01	SOLID	<u>N/A</u>	<u>59.92</u>		%
95000069	C-204	3 DUP	S95T000890	0	TGA-01	SOLID	<u>59.92</u>	<u>56.08</u>	<u>N/A</u>	%

Final page for worklist # 1375

Smilton 5-10-95  
Analyst Signature Date

L. Jones 5-12-95  
Analyst Signature Date

Verified 5/12/95 Jean M. Eyer

Data Entry Comments:

sample is like stiff dark chocolate frosting

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT  
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 3-49 TO 351.

TGA STD 42N8-A

19.976 mg

Rate: 10.0 °C/min

File: 00034.001

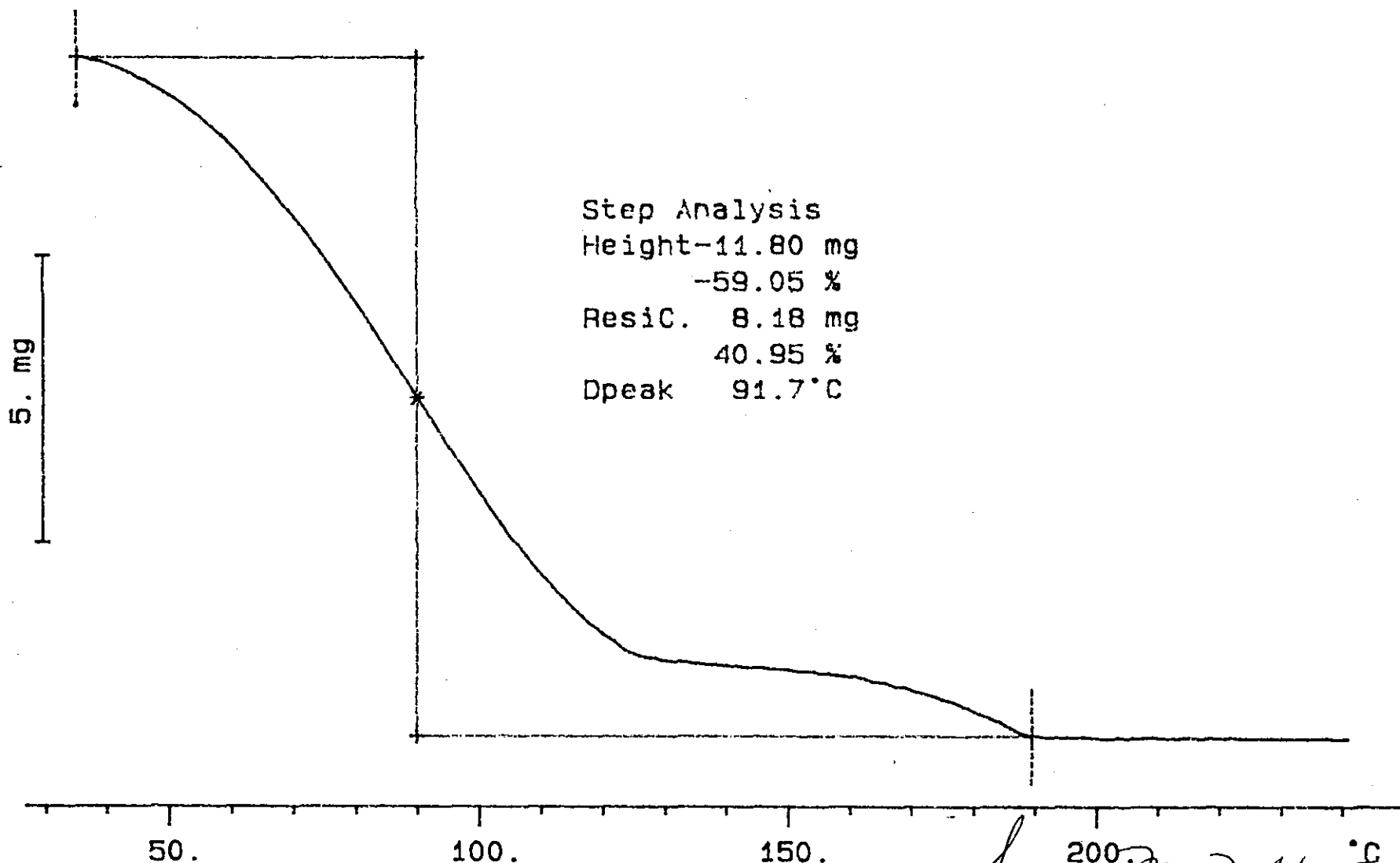
Ident: 0.0

TG

METTLER

10-May-95

222-S Laboratory



3-49

*Susie M. Fulton* 5-10-95

WHC-SD-WM-DB-115, REV. 1  
WHC-SD-WM-DB-115, REV. 1



3-50

S95T000890 N2

26.865 mg

Rate: 10.0 °C/min

File: 00040.001

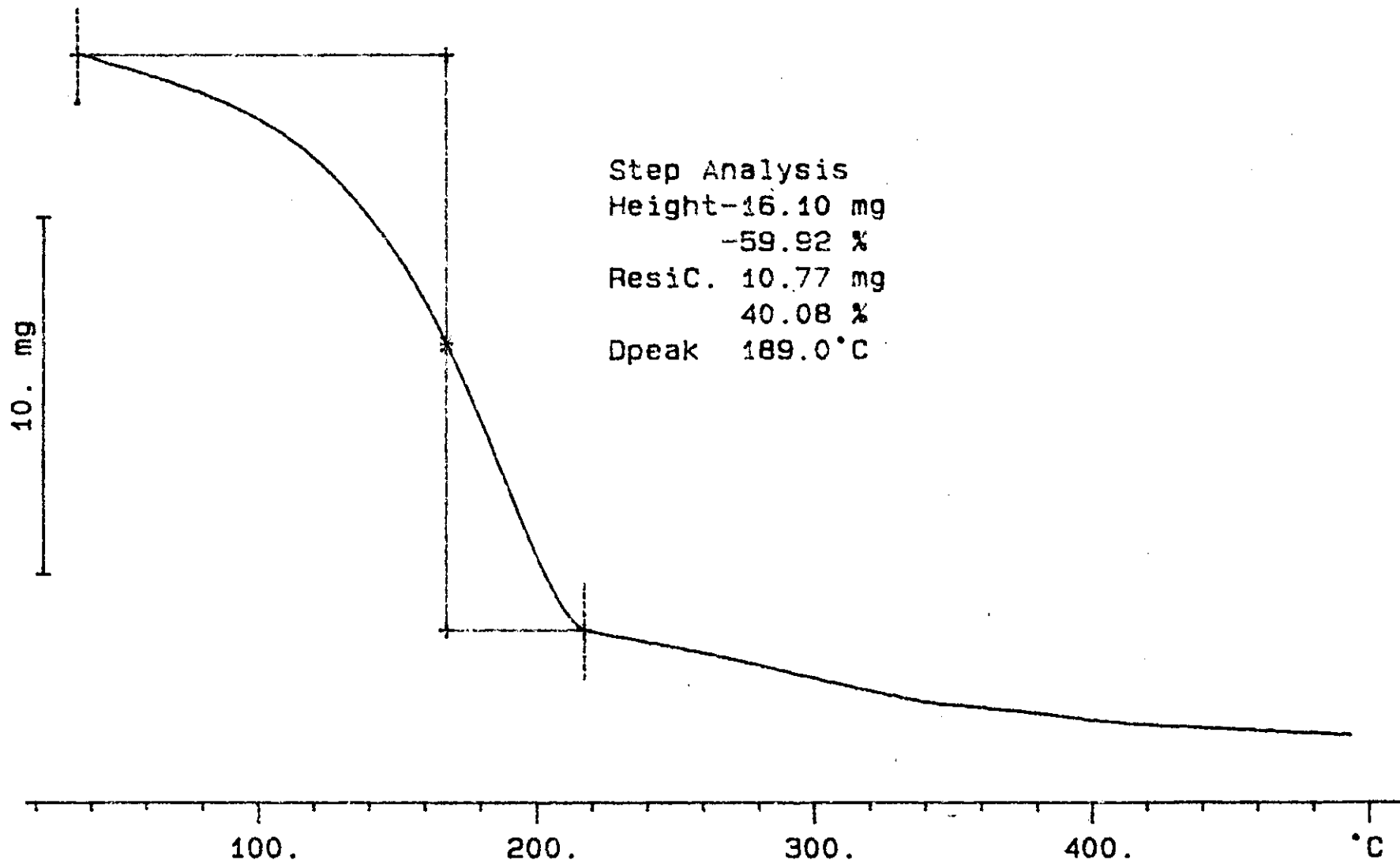
TG

METTLER

10-May-95

Ident: 0.0

222-S Laboratory



3-51

S95T000890 (DUP) N2

27.826 mg

Rate: 10.0 °C/min

File: 00042.001

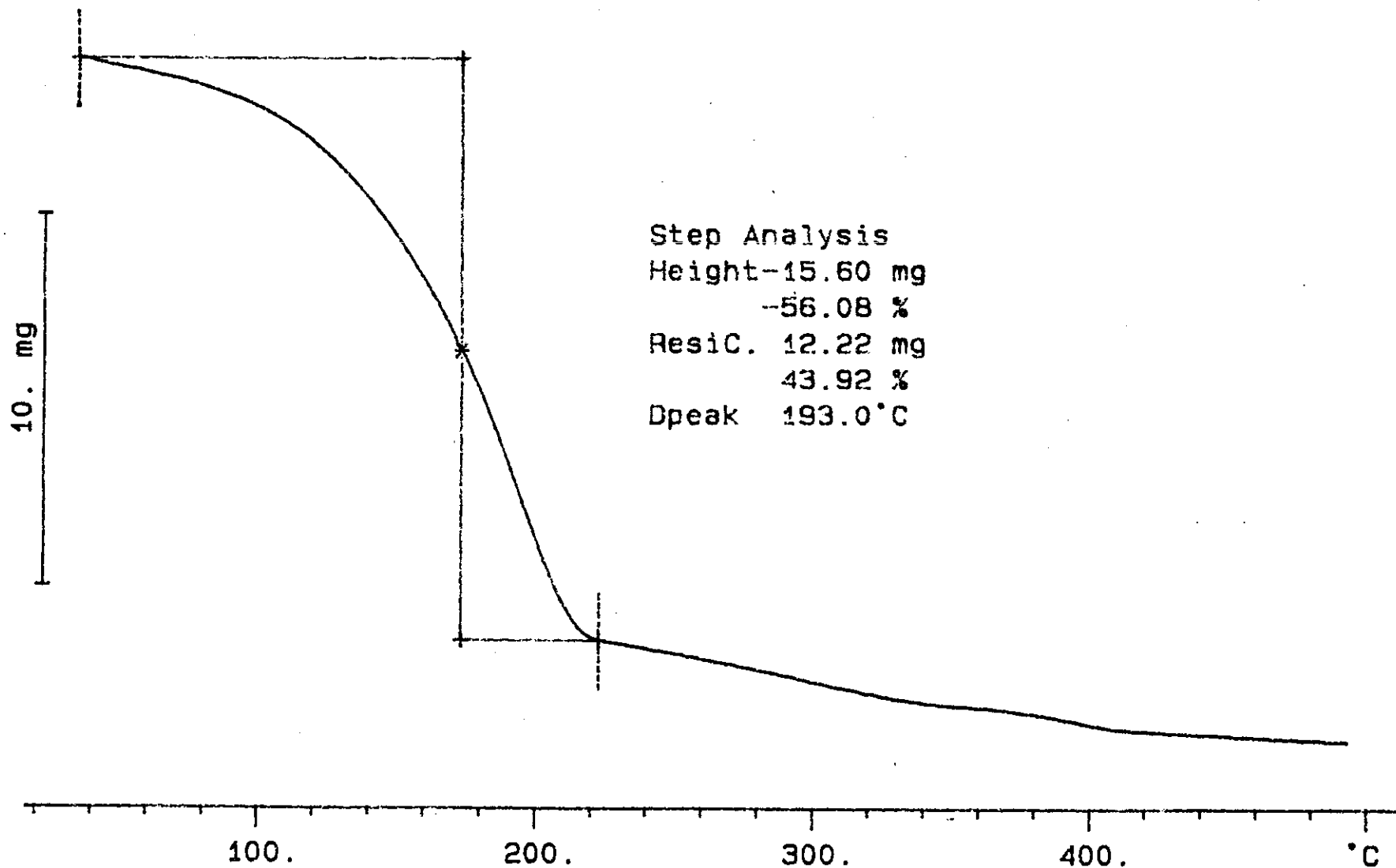
TG

METTLER

10-May-95

Ident: 0.0

222-S Laboratory



WHC-SD-WM-DP-115, REV. 1

WHC-SD-WM-DP-115, REV. 0

# DISTRIBUTION SHEET

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		Date: 07/22/96
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		ECN NO.: 633306

Name	MSIN	Text With all Attach	EDT/ECN ONLY	
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<u>U.S. Department of Energy, RL</u>				
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M. J. Kupfer	H5-49	X		
J. E. Meacham	S7-15	X		
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M. J. Sutey	T4-07	X		
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J. A. Voogd	H5-03		X	
L. R. Webb (SD COV. SHT., DST. SHT, ROR)	T6-06		X*	
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